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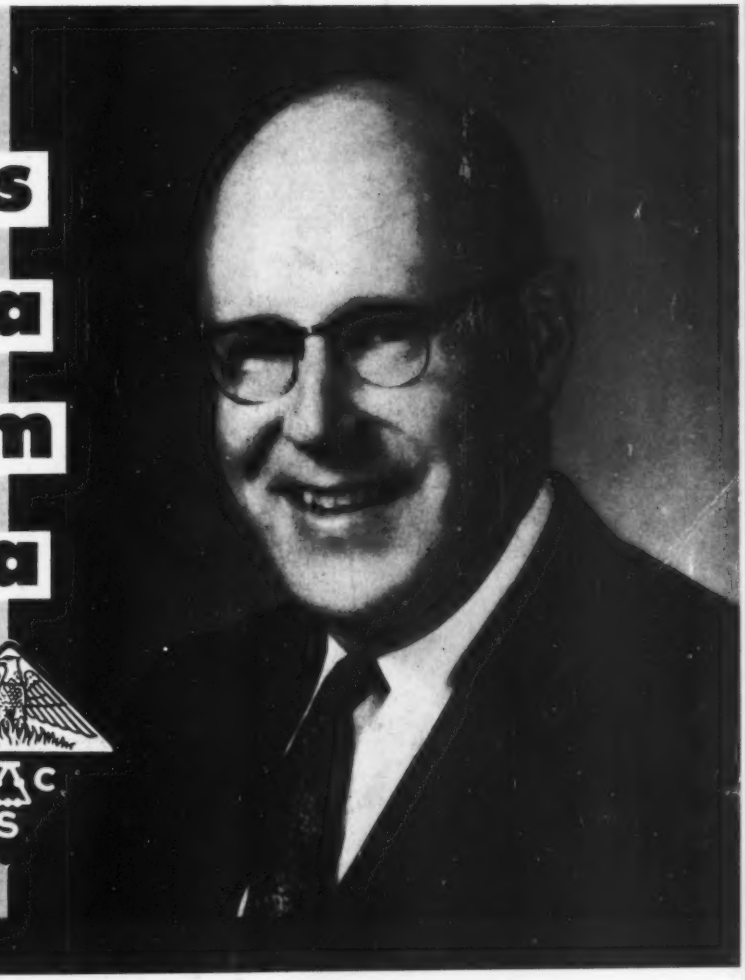

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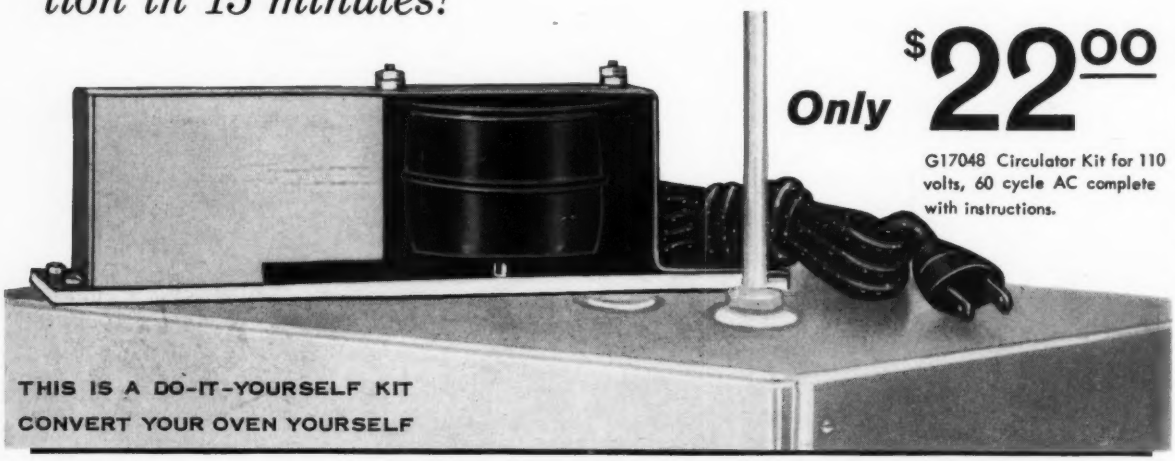

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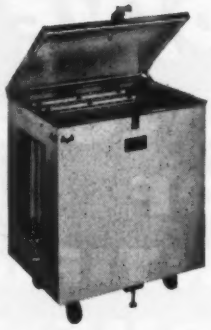

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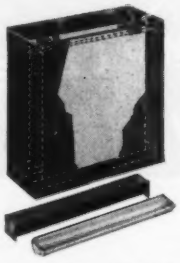

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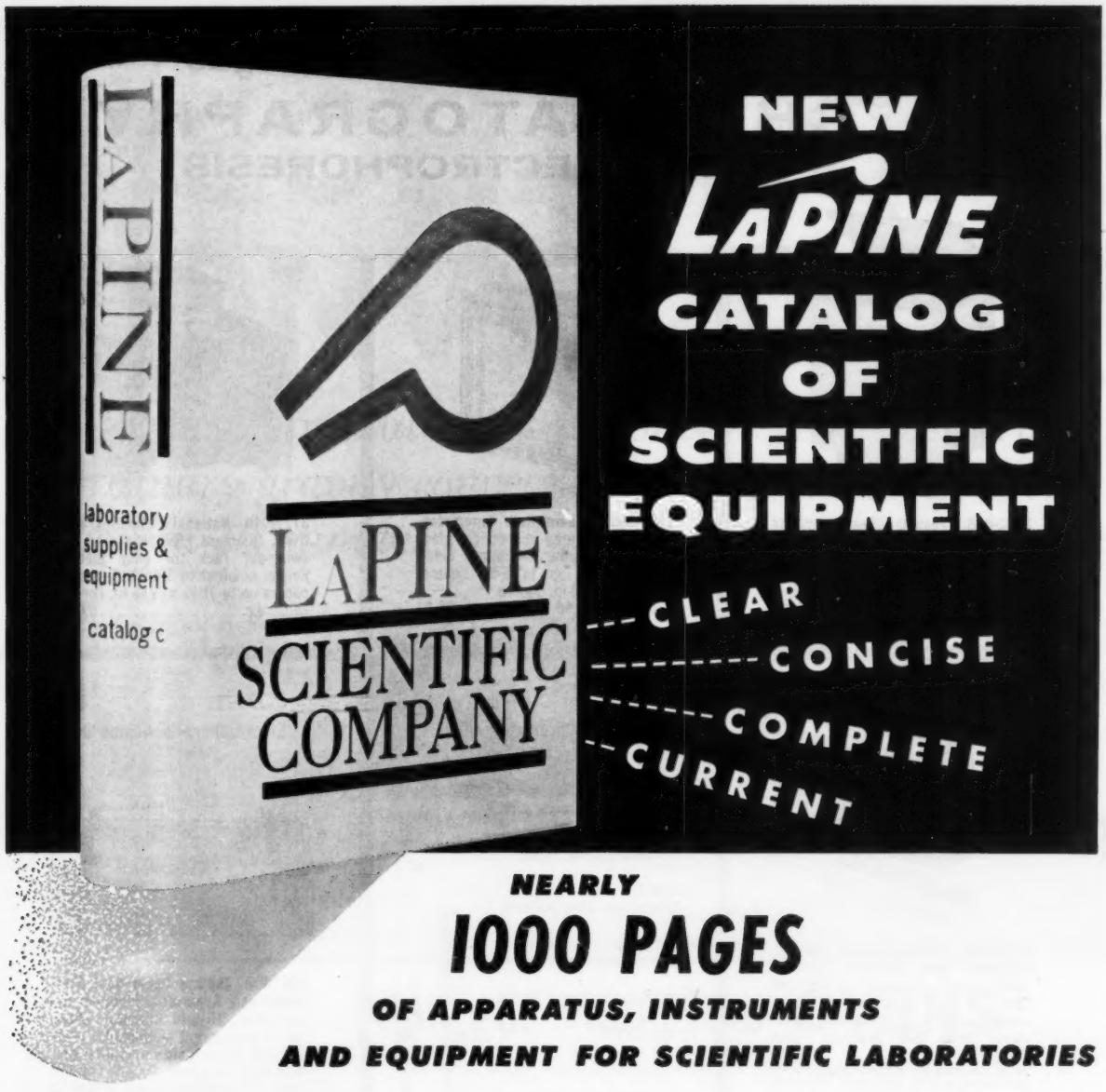

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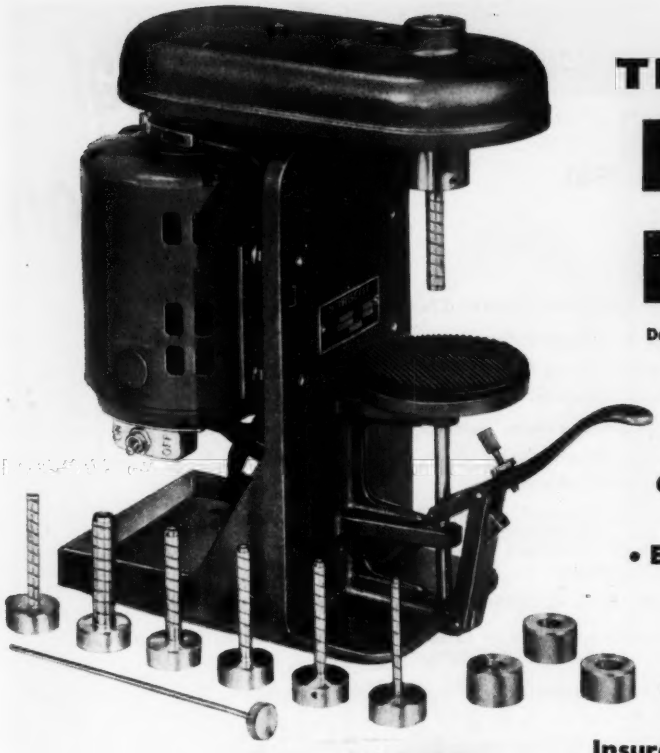

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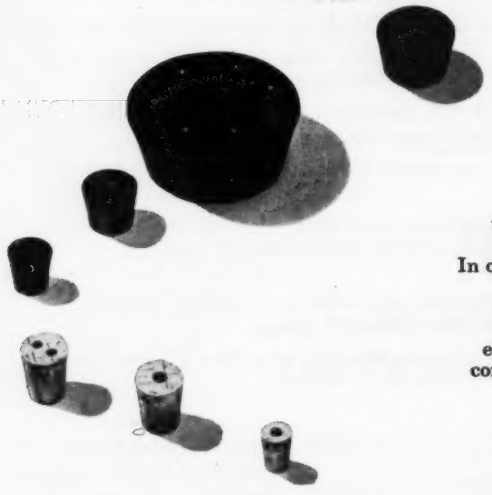

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14. Chromatographic Equipment (Continued)

In column chromatography, the diameter and length of the column container, as well as the particle size and packing of the adsorbent bed, have controlling effects on the sharpness of the separations obtained. Ideally, in an elution chromatogram it would be most desirable if the several components could be resolved completely from each other, with well-defined boundaries for each zone; in a frontal chromatogram one might hope for sharp, almost discontinuous transitions from one front to another. These conditions, which are shown schematically in Figure 14, are of course not observed

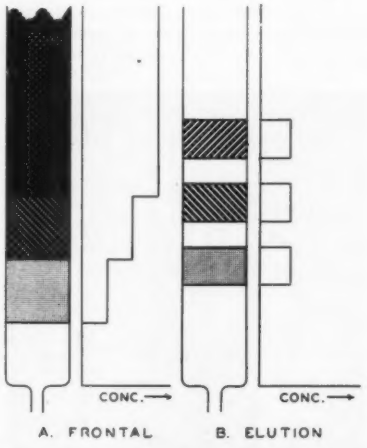

Figure 14. Schematic representation of hypothetically perfect frontal and elution chromatograms, neglecting the nature of the physical phenomena giving rise to the separations.

in practice; illustrations of the characteristic appearance of very good frontal and elution chromatograms are given in Figure 15. It will be noted that the transition between successive fronts becomes less distinct, and the tailing or diffuseness of an elution peak becomes greater, as the retention volume increases. The *retention volume* of a component is the volume of liquid phase that must be passed through the column to bring that component to the bottom (i.e., to cause it to "break through" and become evident in the effluent). Increasing the length of a packed column causes the separation between elution peaks to increase, but the consequent increased retention volume makes the peaks less easy to discern. Increasing the cross-sectional diameter of the column permits larger samples to be chromatographed, but the tendency for the fronts to become irregular and to angle away from the horizontal also increases because of the difficulty of maintaining a high degree of uniformity in a large bed of very small particles. When effluent is collected, the more irregular and non-horizontal the fronts are, the more diffuse and indistinct does the separation of components appear to be, even though the actual separation of these components on the adsorbent itself may be good. This situation is illustrated by the diagrams given in Figure 16.

The degree of sharpness of fronts, as well as of the leading edges of elution peaks, is determined by the physical chemical nature of the adsorption or distribution equilibrium (*cf.* Figures 2 and 3). In most cases, a chromatogram will be better, the closer to equilibrium it is possible to maintain conditions throughout the length of the column. For example, consider a convex distribution curve, such as III in Figure 2. At

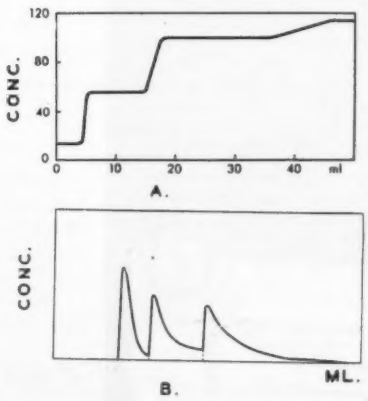

Figure 15. Graphs of concentration versus volume of effluent for very well resolved chromatograms of a three-component sample. A. Frontal development. B. Elution development.

low solute concentrations, the proportion of the component that is extracted into the second or stationary phase (phase 1 in the diagram) is greater than at higher concentrations. Since the rate of travel of the component down the column, under the influence of the flowing phase (phase 2 in the diagram), is slower, the greater the proportion extracted, the component will move down slowly where its concentration in the leading edge of the zone is low, and more rapidly where it is high. Hence, the portion of the zone having a high concentration will tend to overtake and swallow-up the low concentration region of the front, with the result that the leading edge will tend to be sharp and to correspond to a high concentration. The tailing at the back edge of the zone is due to the fact that the amount of component desorbed by fresh solvent moving down

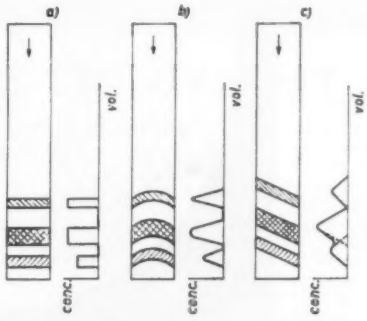

Figure 16. Illustration of the fact that the more irregular or non-horizontal the fronts are within a column, the less well-resolved does the elution chromatogram become.

is always a fraction of what had been adsorbed just before, so that the net amount adsorbed at a given place decreases as a geometrical (i.e., exponential) function of the volume of the eluant.

The various possible shapes of the partition curves (Figures 2 and 3) show that many types of chromatographic behavior are possible, ranging from self-sharpening fronts to fronts that become progressively less sharp with distance of travel.

The achievement of equilibrium conditions in the column is facilitated by the use of an adsorbent of small particle size and high porosity. However, column packings of fine particles have a high resistance to fluid flow, and if the eluant takes too long to pass through the column, much of the sharpness of separation can be lost as a result of the tendency for diffusion to wipe out concentration differences. Consequently, it is common practice in column chromatography with certain adsorbents to use fine particles (e.g., No. 325 mesh) and to apply a pressure of several atmospheres to the top

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of the moving liquid phase to achieve a reasonable rate of flow. Thus, a glass column for this type of work should be made of thick-walled tubing capable of withstanding 3–4 atmospheres (*cf.* Figure 10).

The advantages of the use of columns packed with adsorbent stem principally from the flexibility these offer with respect to the composition of the two-phase system (i.e., choice of adsorbents and eluants), and the possibility of adjusting the operating conditions (e.g., column dimensions, flow rate) to the characteristics of the sample being analyzed. A packed column is most appropriate if semimicro or micro scale samples are to be worked up. The disadvantages of packed columns include the necessity of either extruding and carving up the adsorbent bed in order to separate the zones of components, or of collecting, evaporating, or otherwise analyzing large volumes (or a large number of small volumes) of effluent. Column chromatography is often very time-consuming and tedious. The location of the various (non-colored) components within the column is generally not susceptible to test, and it is not possible to run a standard sample side-by-side with an unknown on a given column.

As a consequence of the manipulative inconvenience of column chromatographic procedures, much effort has been devoted to the development of alternative, more attractive techniques. The most successful of these techniques is *paper chromatography*, in which a strip or sheet

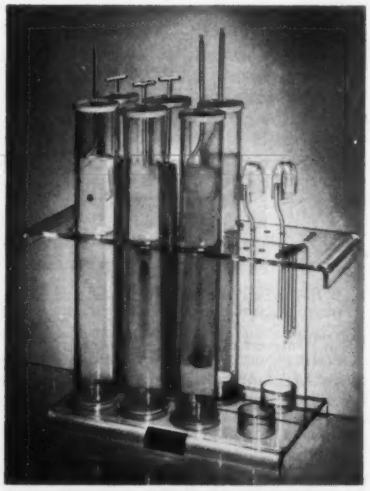

Figure 17. The Gordon-Misco multiple tube equipment of Microchemical Specialties Co. for paper strip chromatography.

of paper, composed of cellulose or glass fibers, and, if desired, impregnated with ion exchange resins, serves as the solid phase. The paper may function as a surface-active phase (*adsorption chromatography*), or merely as a supporting substrate to immobilize a film of a liquid

(*Continued on page A571*)

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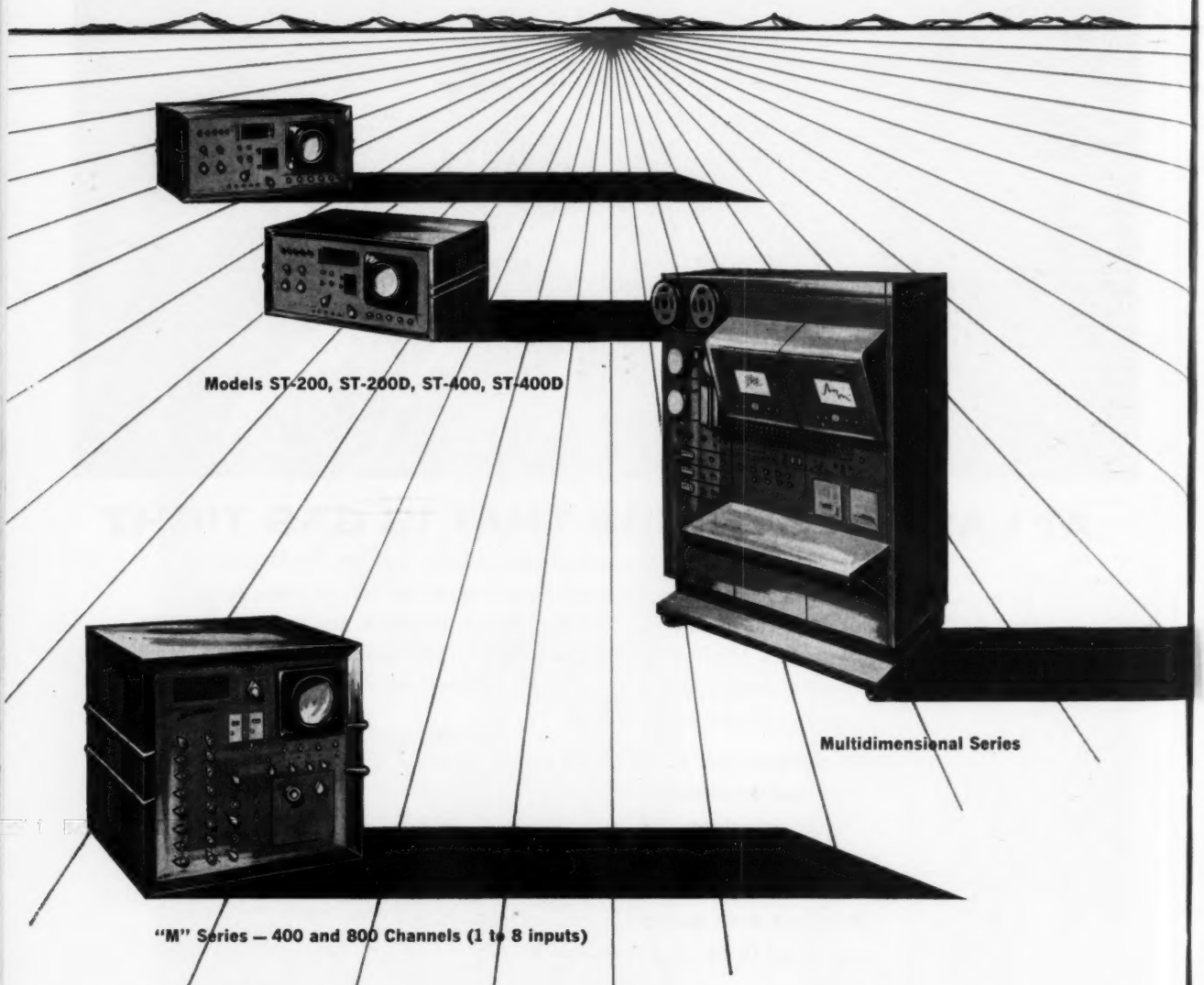

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(*partition chromatography*), or it may combine these functions as well as some degree of ion exchange activity.

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Ascending Paper Chromatographs

For ascending paper chromatography, it is only necessary to mount the paper strip or sheet so that it hangs vertically, with its bottom dipping into a reservoir of the developing liquid. In order to prevent irregular and irreproducible flow patterns and temperature inhomogeneities, the paper must be contained in a sealed volume which is saturated with the vapor of the developing liquid, so that there is no tendency for vapor to distill from the wetted portions of the paper. If the wet paper touches its container at any

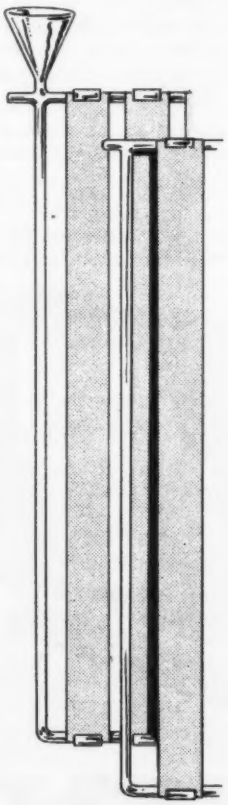

Figure 18. Kurtz-Miramon frame of Kensco for ascending paper strip mounting.

point except where it is in contact with the pool of liquid on the bottom, siphoning of liquid will occur, and the chromatographic development will be vitiated.

A number of containers and mounting

(*Continued on page A572*)

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accessories are commercially available for ascending work, ranging from simple, stoppered tubes for single strips, to large battery jars capable of holding a hundred or more strips for simultaneous development.

A convenient six-tube unit for multiple simultaneous experiments which is available from Microchemical Specialites Co., Berkeley 10, California (Gordon-Misco Tube Chromatography Laboratory, one-dimensional ascending, $78) is shown in Figure 17. Each tube consists of a Pyrex cylinder open at both ends, fitted with polyethylene stoppers. A solvent cup with sloping sides, so that the paper strip can dip into the liquid without touching the sides, sits on the lower stopper, and a clip holder for the paper strip is mounted in the upper stopper. Special cups are also available for attachment to the upper stopper so that these units can be used for descending chromatography (6 cups, $20).

Polyethylene or Teflon clamps are employed for mounting the paper strips on a glass frame in the apparatus made by Kensington Scientific Corp. (Kensco), Berkeley 10, California, formerly California Laboratory Equipment Co. (Calab), as illustrated in Figure 18. The funnel attached to the vertical tube is used for adding solvent to the bottom of the container. Standard units are available consisting of a glass tank with close-fitting cover, a rack for holding 20 frames each of which carries up to 7 one-inch wide paper strips, and 280 polyethylene clamps (No. 1007, $159).

A container for ascending chromatography with square paper sheets, so that

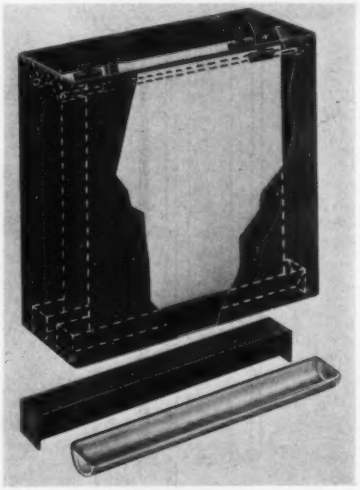

Figure 19. Kensco stainless steel tank for ascending paper chromatography.

two-dimensional separations can be carried out by employing two runs with different solvents, the paper sheet being rotated by 90° between runs, is illustrated in Figure 19 (Kensco No. 1013 Stainless Steel Tank, $41.20). The tank dimensions are chosen to provide for the mounting of two 8 × 8-inch wide sheets, with a small

(Continued on page A574)

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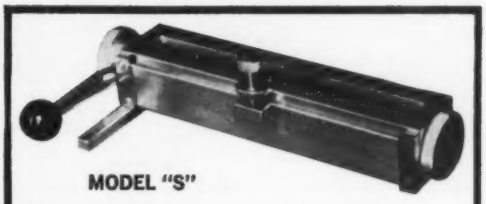

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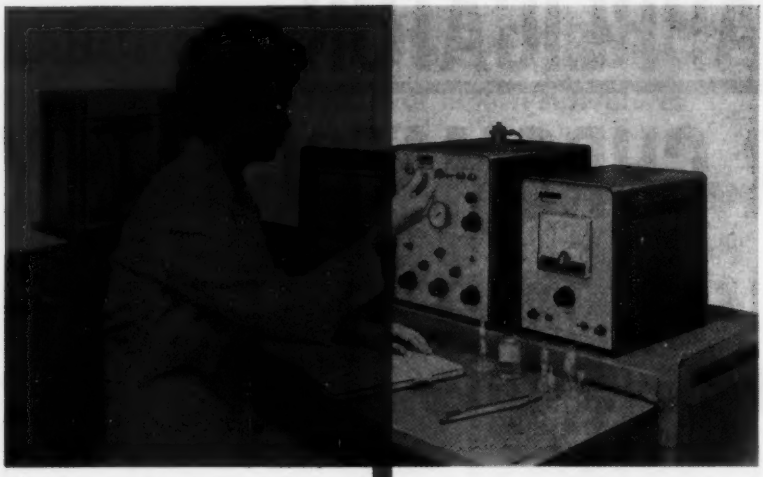

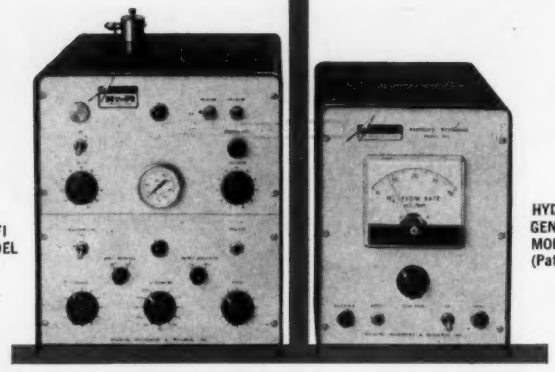

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free tank volume, so that vapor equilibration is rapidly achieved. The paper sheet is held at the top by means of stainless steel spring clips to stainless steel or glass rods. The tank is covered with a flat plate glass cover to permit observation while preventing loss of vapor. In some cases, a specimen migrates up the paper very slowly, and it may be desired to continue the ascending chromatogram for long periods. For this purpose, a cover glass with narrow slits may be employed to seal the tank; the paper sheets are cut long enough to permit them to stick out of these slits. When the mobile phase reaches the top of the tank, it evaporates from the paper, and fresh liquid continues to mount up the paper to replace that which is lost by evaporation.

A large tank constructed entirely of $^3/_8$-inch thick polyethylene, with a plate glass viewing top, and capable of mounting eight 17 × 17-inch sheets for ascending chromatography is available from Palo Laboratory Supplies, Inc., New York 7, N. Y. (Chromatography Bench Unit, Model 7700, $154).

A novel approach to the design of a container for paper strip chromatography is embodied in the Chromatobox of Research Specialities Co., Richmond, California, shown in diagrammatic form in Figure 20. A paper strip, one inch wide and up to 17 $^5/_8$ inches long is placed on

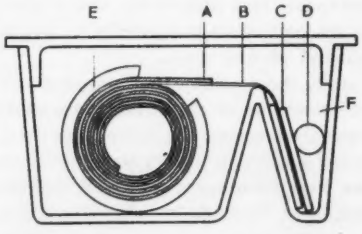

Figure 20. The Chromatobox, manufactured by Research Specialties Co. *A.* Teflon band. *B.* Paper strip. *C.* Glass plates. *D.* Glass rod. *E.* Holding ring. *F.* Solvent chamber.

a Teflon strip, and the pair is rolled up into a coil. The Teflon has an embossed surface of many small raised dots, as shown in Figure 21, so that there is relatively little actual contact with the paper strip; in addition, the non-wettability of Teflon by most solvents helps to insure that the sample and solvent will travel along the paper, and will not leak off onto the plastic. The coil assembly is placed in a molded plastic box, with the free (wick) end of the paper anchored between glass plates and dipping into a small solvent compartment. Because of the small size of this unit, saturation of the enclosed atmosphere and development of the chromatogram require only 6–8 ml of solvent (Model A-801, $10).

Descending Paper Chromatographs

The design of chromatographs for the descending technique differs from that

(*Continued on page A576*)

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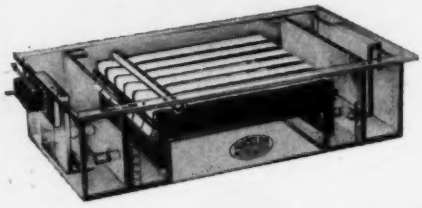

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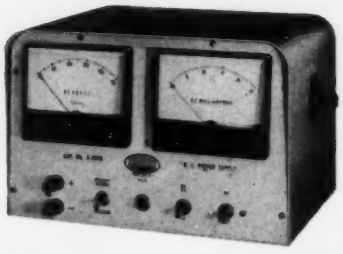

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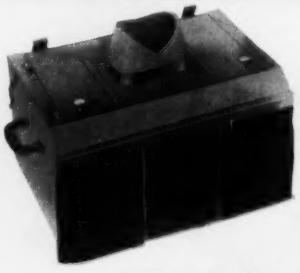

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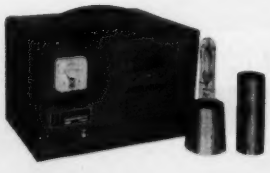

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employed for ascending work only in the requirement for a reservoir of solvent to be placed at the top of the paper sheet, rather than at the bottom. The typical

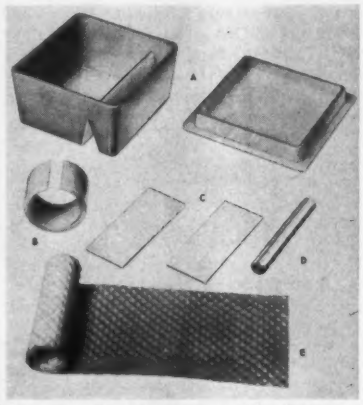


Figure 21. Component parts of the Chromatobox. *A.* Box and lid. *B.* Holding ring. *C.* Glass plates. *D.* Rod. *E.* Teflon Band.

construction of such a reservoir is shown in Figure 22. The paper is held in a solvent trough by the weight of a glass anchor rod. Precautions must be taken to prevent liquid from siphoning out of the trough by climbing along the paper to the rim of the trough and then running down its underside. This is avoided by

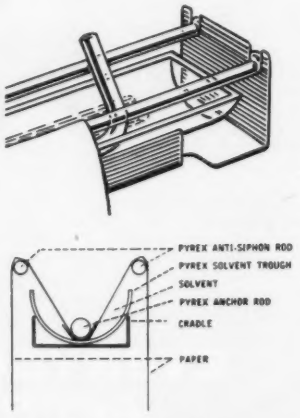


Figure 22. Detail of the design of a typical solvent reservoir and paper support for descending paper chromatography.

making the paper rise up some distance above the trough before it changes direction and falls to the floor of the container; the anti-siphon rods provide the required support to hold the paper away from the trough.

Figure 23 shows a hydrometer jar apparatus made by Kensington Scientific Corp. for descending or ascending paper sheet or strip chromatography (No. 1015, $118). It consists of a stainless steel holder for the paper and solvent, and a Pyrex hydrometer jar, $4\frac{1}{2} \times 24$ inches,

with a ground flat top and a stoppered plate glass cover. For descending work, solvent is placed in the upper trough; for ascending work the solvent is introduced through the hollow center rod into a trough at the base. After insertion of the paper inside the wire struts, the application of the sample, chromatographing, drying, and staining can all be carried out without touching or handling the paper.

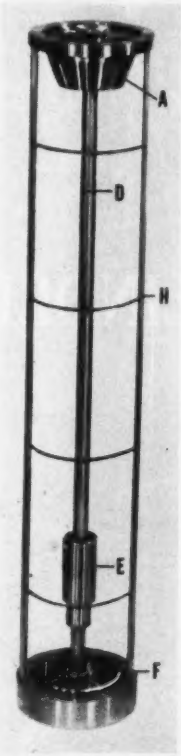
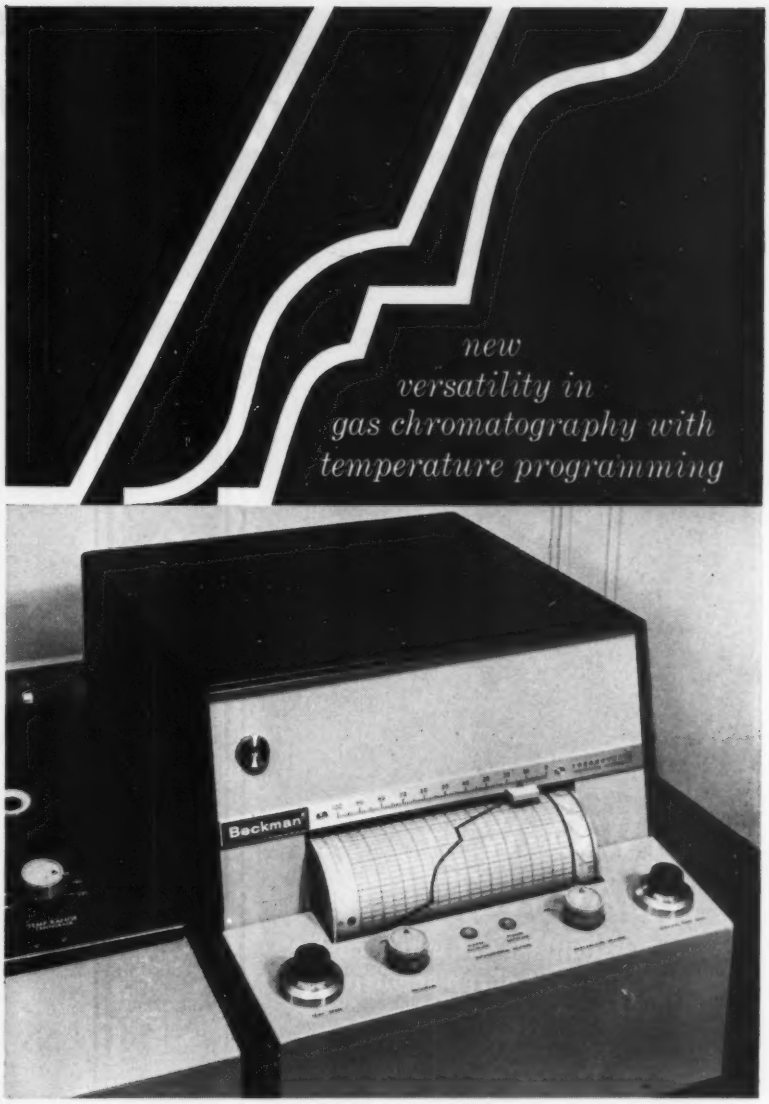


Figure 23. Kensco hydrometer jar apparatus for ascending or descending paper chromatography. *A.* Removable upper trough. *D.* Hollow center rod. *E.* Center receptacle for solvent which feeds paper roll slipped over center rod to aid in vapor saturation of enclosed space. *F.* Base trough. *H.* Wire struts.

Containers for paper chromatography are most generally made of glass or of stainless steel. The metal constructions are more compact and rugged, and permit the solvent trough to be mounted on brackets bolted to the upper walls. However, stainless steel is corroded by halogen-containing substances, such as HCl, and its use should be avoided when solvents containing such substances are to be employed. Glass containers have the advantage of being resistant to all the solvents used in chromatographic work, but the mounting of a solvent trough presents problems. One approach is the use of an annular ledge near the top of a glass cylinder as a support for the trough (see Figure 24), or an all-glass support rack reaching from the bottom to the upper portions of the cylinder.

(Continued on page A580)



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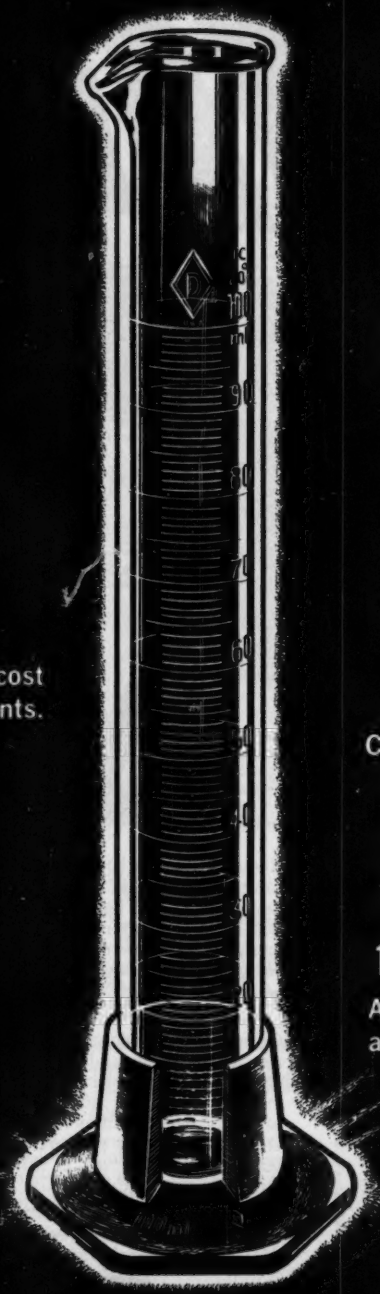

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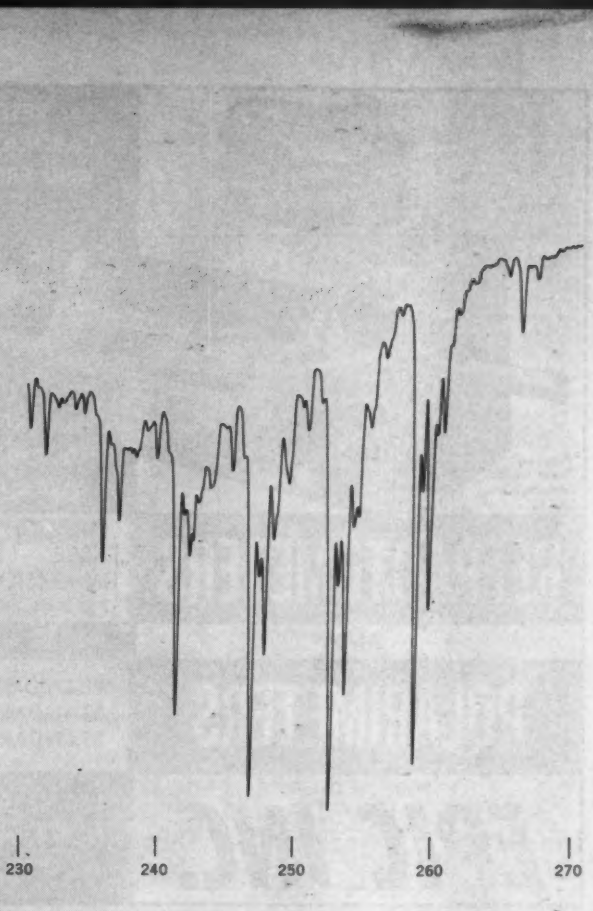

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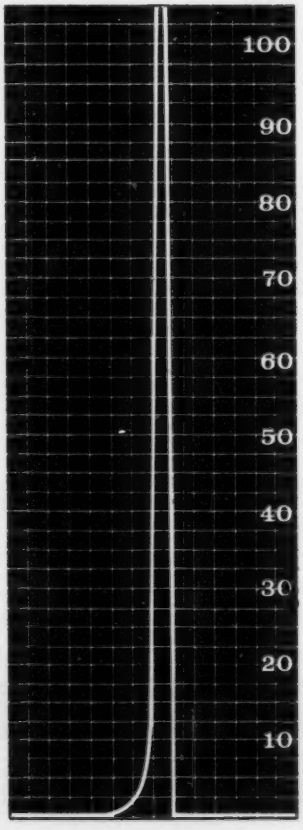

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Chemical Instrumentation

When some stainless steel parts can be tolerated, band-spring supports, or metal rack constructions are used (see Figure 25).

Figure 24. All-glass apparatus for descending paper chromatography. Solvent trough is mounted by resting on an annular ledge.

Horizontal Paper Chromatographs

Several convenient devices have been proposed for horizontal paper chromatography with circular discs. A commercially available design that permits the rapid comparison of unknowns and

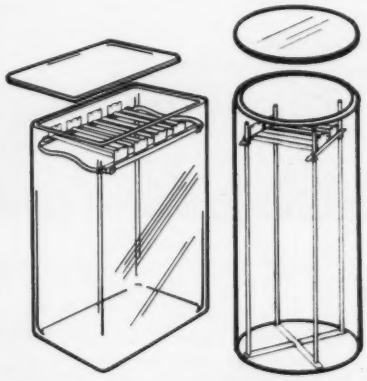
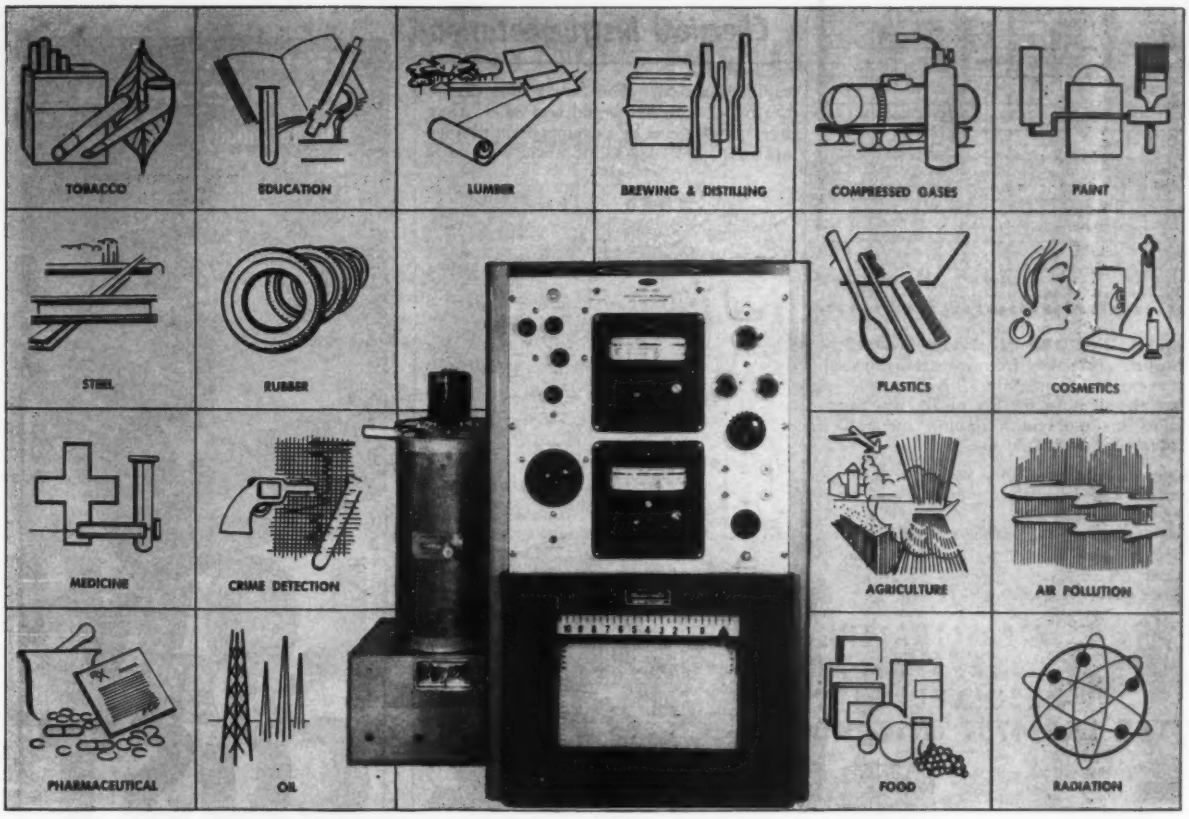


Figure 25. Upper solvent troughs may be held in place in glass apparatus by means of stainless steel spring bands, or by a rack construction.

standards is the Kawerau apparatus, shown in Figure 26, which is made by the Shandon Scientific Co., Ltd., London; U. S. distributor, Consolidated Laboratories, Inc., Chicago Heights, Illinois (No. 2020, 14-inch diameter unit, $28.20). A slotted paper disc is placed with its center resting on the top of a glass capillary tube which dips into a solvent reservoir. This capillary serves as a wick to feed solvent to the center of the paper, from which point it spreads out radially by

(*Continued on page A582*)



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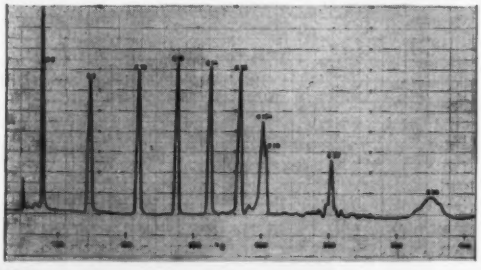
MODEL 500 LINEAR PROGRAMMED HIGH TEMPERATURE GAS CHROMATOGRAPH

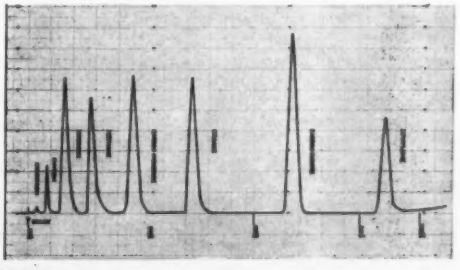
The technique of linear programmed temperature gas chromatography combined with high temperature operation makes possible these previously difficult analyses: (1) wide boiling mixtures; (2) gases (H_2, O_2, N_2, CO, CO_2 and light hydrocarbons); (3) trace quantities of high boiling residues in low boiling solvents; (4) correlation of retention times with analytical distillation data; and (5) temperature sensitive materials.

Two such analyses are shown below. Note that with programming, both separations required only one column. Without programming, the methyl ester separation would have required a fixed high column temperature with a loss in resolution of the lower boiling components. In the separation of gases at a constant column temperature, ethane, carbon dioxide and ethylene would not have eluted. To chromatograph this mixture at constant temperature, two columns and two samples are necessary.

With a Model 500, column scouting is easily performed because columns can be changed and baselines re-established in ten minutes. Heating from room temperature to 300°C takes about 10-15 minutes (27 minutes to 500°C) and cooling back to room temperature takes about the same time. Model 500 has separate and independent temperature controls, each to 500°C, for column, detector, and injection port. In addition, eighteen linear heating rates from 0.29 to 42°C/min. provide a wide selection for optimum column conditions.



CONDITIONS:
COLUMN TYPE: 2 FT. SILICONE GUM RUBBER
COLUMN TEMP. PROGRAMMED AT 5.6°C/MIN. FROM 75°C to 400°C.
This is a typical programmed temperature chromatogram demonstrating a separation of methyl esters with widely differing boiling points. Observe the sharp peaks, shaped nearly alike.



CONDITIONS:
COLUMN TYPE: 4 FT. MOLECULAR SIEVE 5A
COLUMN TEMP. PROGRAMMED AT 4.0°C/MIN. FROM 35°C TO 247°C.
By programming column temperature, H_2, N_2, O_2, CO, CO_2, ethane, and ethylene all separate on a single column in one run. Using the same column, the programmed temperature technique works equally well for the separation of nitrous and nitric oxide in the presence of these gases.

Similar analytical advantages with Model 500 apply to the other industries illustrated above. Write for our bulletins on Model 500. These describe in detail all the specialized analyses.

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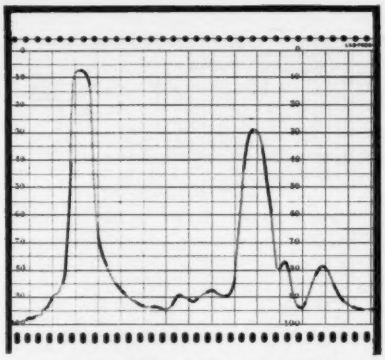
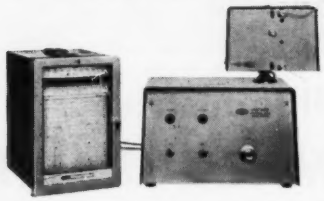


THIS UVICORD DIAGRAM SHOWS:
Peptides released from α-casein through enzymatic degradation by action of Lactobacillus lactis. Six peaks are separated by molecular sieving through a column of SEPHADEX G50.

Column: Sephadex G50, 2 × 110 cm
Sample: 180 mg mixture of peptides with varying molecular size
Buffer: glycine-0.1-N sodium hydroxide, pH 9.9
Flow rate: 30 ml/h at 0.3 atm. air pressure on buffer
Chart speed: 10 mm/h

* A FASCINATING SEPARATION RESOLVED WITH THE LKB 4701 UVICORD



Connected directly to the column outlet, the *Uvicord* measures continuously and without appreciable distortion the UV absorption of an eluate, revealing the interesting components even when present in small quantities.

*** CHARACTERISTICS**

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Five measuring cells of very small volume are available:

Optical Path Length	1 mm	2 mm	3 mm	4 mm	5 mm
Volume of Cell	0.05 ml	0.1 ml	0.1 ml	0.2 ml	0.3 ml

HIGH SENSITIVITY AND WIDE MEASURING RANGE
Example 1. 0.00036 % thyrosine in 0.1-N NaOH, 5 mm cell ≃ 10 mm pen deflection (E = 0.05)
0.036 % thyrosine in 0.1-N NaOH, 1 mm cell ≃ 100 mm pen deflection (E = 1)
Example 2. 0.02 % serum albumin in 1-% NaCl, 5 mm cell ≃ 10 mm pen deflection (E = 0.05).

LOW UV DOSE ON SENSITIVE MATERIAL
Absorbed UV energy is approx. 1 milliwatt-second per ml at 10 ml/hour. This corresponds to a deterioration factor in bovine serum albumin of only 0.0001.

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Drift is less than 2% (transmission) in 24 hours and less than 5 % for room-temperature changes of 15°C, either from 15 to 30 °C, or with coldroom attachment from 0 to 15 °C.

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capillary action. The radial slots divide the paper into five equal sectors, so that five samples can be developed simultaneously.

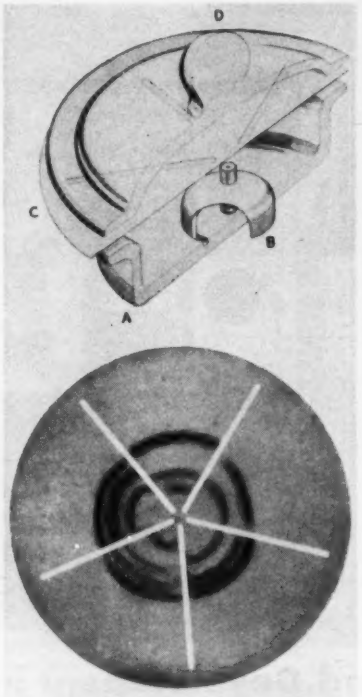

Figure 26. Kawerau apparatus for horizontal paper disc chromatography. Upper drawing shows construction details; lower shows a typical chromatogram. A. Dish. B. Glass support for capillary tube. C. Slotted paper circle. D. Cover. Lower photo: Typical paper disc chromatogram with 5 different specimens.

Centrifugally-Accelerated Chromatographs

An ascending chromatogram may require hours to days for adequate resolution of the components of a mixture. A descending chromatogram is considerably faster, because the flow of developer is accelerated by the pull of gravity. Instruments have been designed in which the force of gravity is replaced by centrifugal force, and by this means chromatograms which would require several hours by the descending technique can be completed in minutes. A schematic diagram of the Chromatofuge ($1295) of Labline, Inc., Chicago 22, Illinois, is shown in Figure 27.

A circle of filter paper, up to 18 inches in diameter, is fitted onto a shaft that is rotated by a variable-speed motor (up to 900 rpm). It is held in place by a Teflon gasket, and is supported on an array of metal pins. While the paper is whirled, solvent is fed onto the sheet continuously through a capillary feed tube mounted close to the rotating shaft. The solvent flow rate is controlled by the diameter of the capillary, and a set of interchangeable capillary tubes is provided to permit

(*Continued on page A584*)

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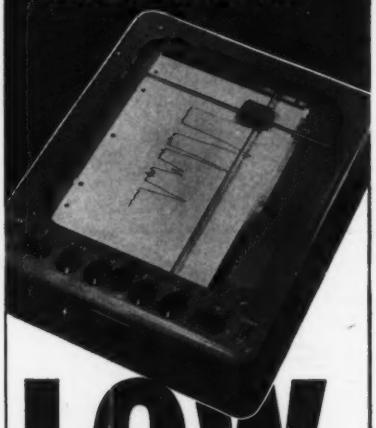

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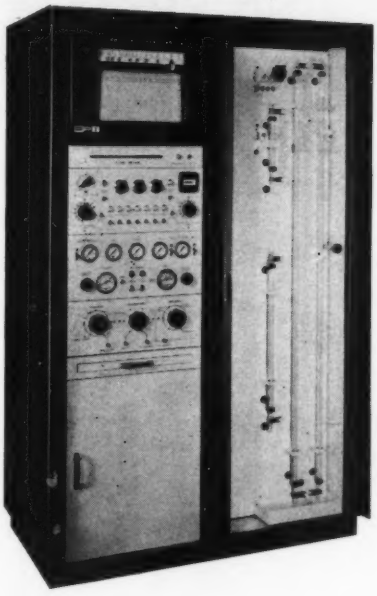

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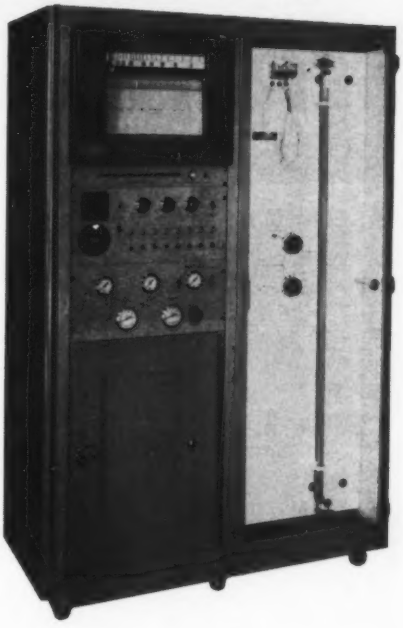

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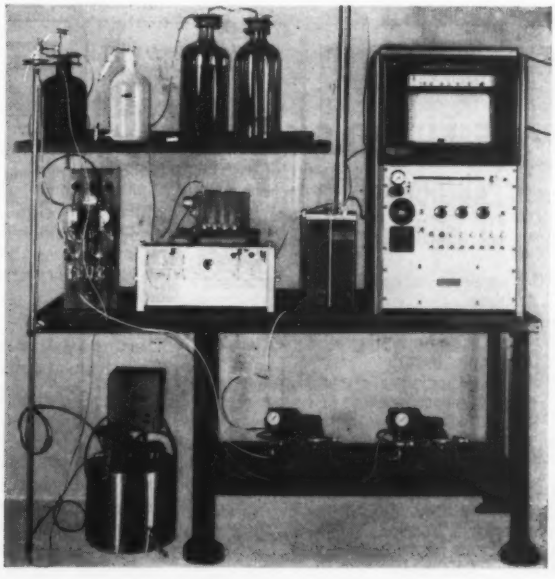

MODEL 5400

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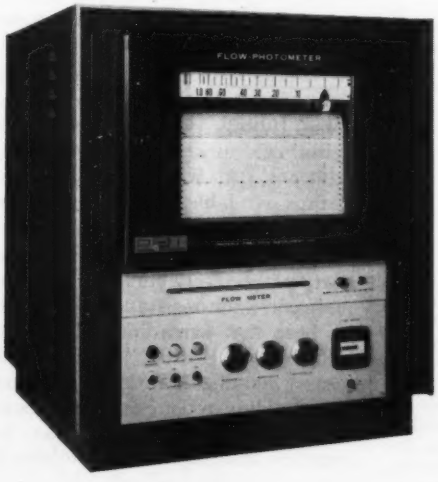

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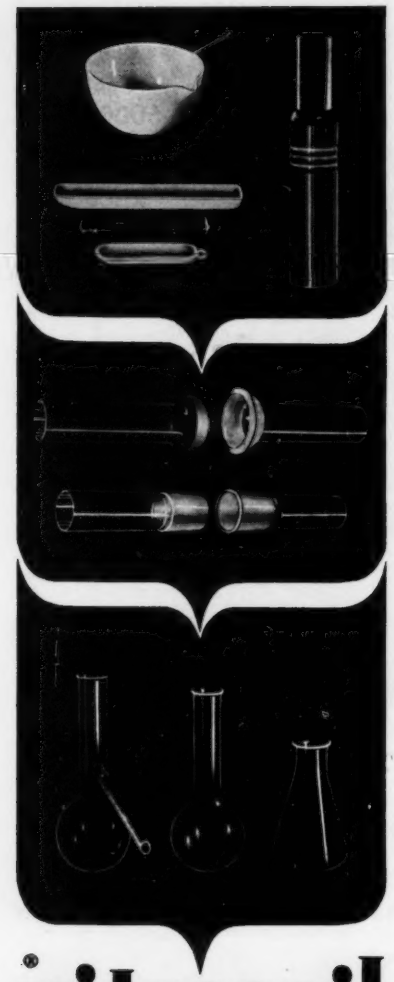

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a choice of flow rates. Centrifugal force causes the solvent to flow radially outward, producing chromatographic development of any specimens which had previously been placed on the paper. Special provision is made for saturating the enclosed atmosphere with solvent and preventing loss of solvent vapor during the centrifugation. Reproducibility of migration velocities is aided by the incorporation of fast starting and braking mechanisms, and the speed of rotation is shown on a continuous-reading tachometer.

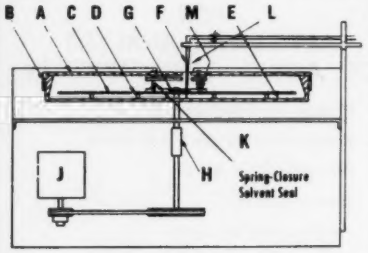

Figure 27. The construction details of the Chromatofuge manufactured by Labline, Inc. A. Cover. B. Collector trough. C. Filter paper circle. D. Support pins. E. Rotating head. F. Solvent feed pipet. G. Hold-down nut. H. Drive shaft and bearing assembly. J. 1/3-hp variable speed drive. K. Spring closure seal. L. Short bore capillary feed tube. M. Teflon sealing ring.

A smaller and simpler version of this type of apparatus is manufactured by Precision Scientific Co., Chicago 47, Illinois (Hi-Speed Chromatograph, $250). A direct-drive ac motor is employed, with a choice of rotation speeds between 300 and 1500 rpm. Solvent is fed onto the paper through a 0.1-mm orifice in a stainless steel nozzle, and the rate of flow is controlled by adjusting the air pressure over the solvent in a reservoir that feeds the nozzle. Paper circles up to 12½ inches in diameter can be accommodated. An explosion-proof version of this instrument ($275) is available, in which an air-driven motor is used, to avoid any possibility of a spark from an electric motor igniting solvent vapors which may be present in a chromatography laboratory.

Thermally-Accelerated Chromatographs

The migration velocity of many constituents under the influence of a moving liquid phase can be markedly increased by raising the temperature, and efforts have been made to speed up chromatographic development techniques by operating at elevated temperatures. This has not proved successful in the standard ascending or descending techniques because of the difficulty of maintaining uniform and constant conditions throughout the system. However, a technique based upon a horizontally supported paper sheet contained in a flat, sealed tray which is kept in a thermostatically-controlled

(*Continued on page A586*)

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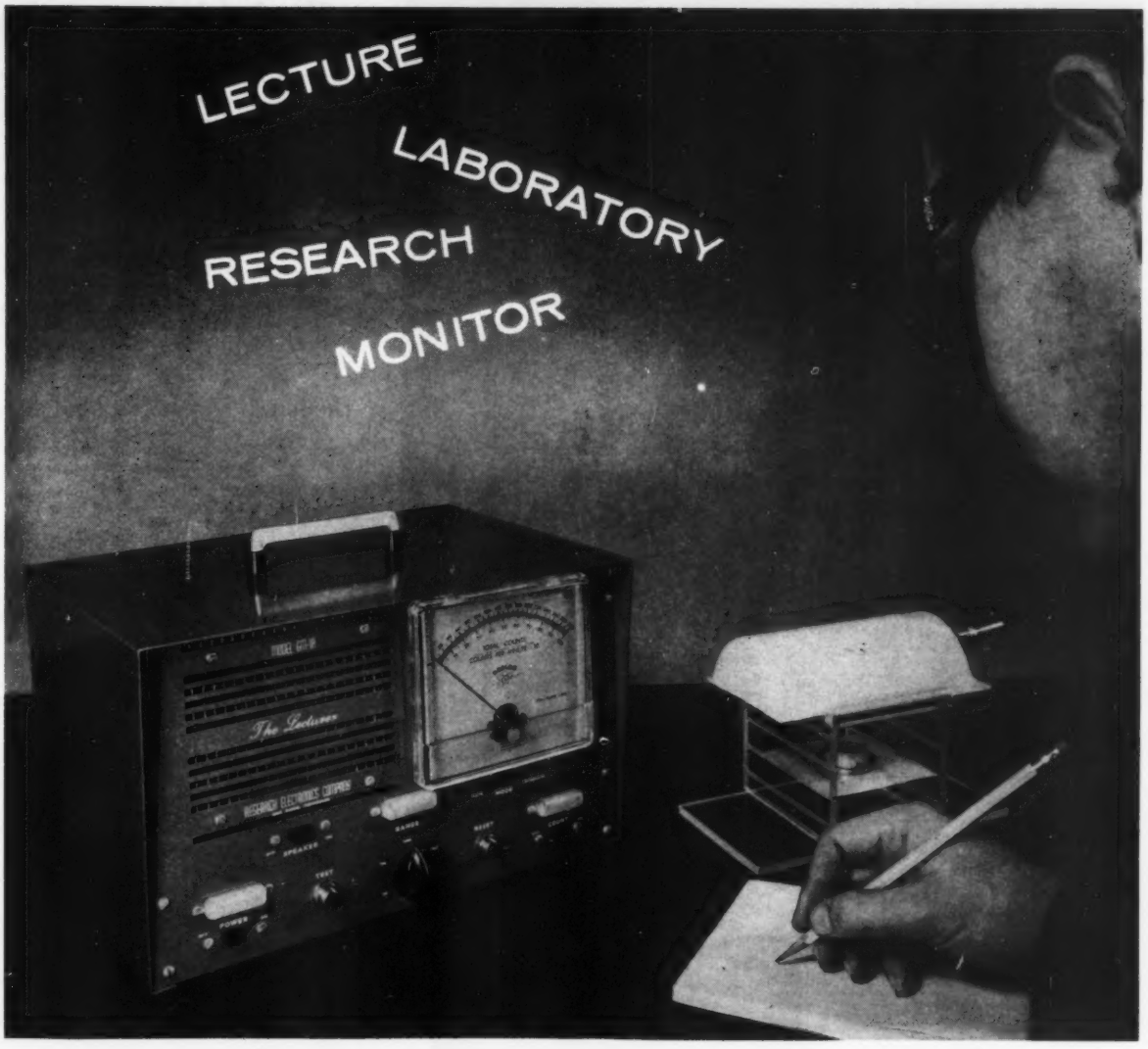

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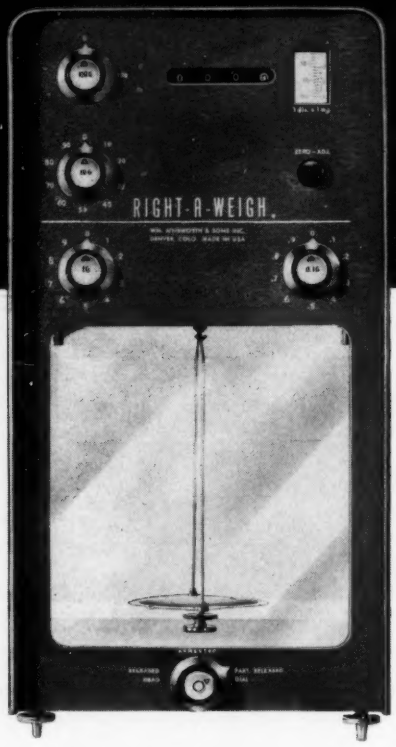

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oven has been found to succeed in achieving adequate separations in 2 to 3 hours for constituents which require 48 to 96 hours by the room-temperature ascending technique. The method of mounting the paper is illustrated in Figure 28. One end of the paper sheet is bent down and inserted in a solvent

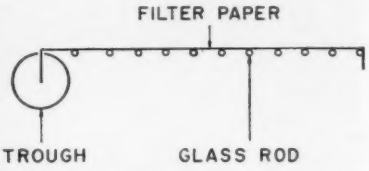


Figure 28. Arrangement of paper and feed trough in the Speedi-Cell elevated temperature migration chamber.

trough; the sheet is supported on a series of glass rods, and the other end is serrated to produce a series of drip tips, and then is bent down to allow the solvent to drip off after it has traveled the length of the paper. Thus, a continuous flow of the developer can take place. The trough, paper, and glass rods are contained in a shallow stainless steel tray (9 × 13 inches) which is covered with a Tygon-gasketed plate glass cover, held on with C-clamps. The tray and its contents are placed on a shelf of an oven maintained at 60°C and left there for the desired period of time. These migration chambers are made by Labline, Inc., Chicago 22, Illinois (Speedi-Cell, $70).

Electrolytically-Accelerated Chromatographs

Still another approach to providing a motive agency other than capillary action for causing differential migration along a paper sheet is the use of an electric field. Since electromobility is a specific molecular property that can by itself be made to yield a selective distribution of components, the application of a potential to a wetted paper strip can result in a chromatographic development that is a function of (*A*) electromobilities, (*B*) adsorbabilities, and (*C*) distribution (partition) coefficients. The relative importance of these several factors in the over-all result depends upon the solvent system, the nature of the sample, and the design of the apparatus.

McDonald has coined the term "ionograph" for an instrument designed so that the analytical separation obtained is due principally to the specificity of electromigration, and effects due to solvent flow or partition between a mobile and a fixed phase are minimized. That is, the results of an experiment with an Ionograph would be equivalent to those of free electrophoresis in a liquid, the paper or other solid phase present in the apparatus only serving mechanically to stabilize the liquid. McDonald's device is shown schematically in Figure 29;

(*Continued on page A588*)

ANALYTICAL and PREPARATIVE METHODS OF SEPARATION

GEL ELECTROPHORESIS

Electrophoresis on gel supporting media affords much improved resolution in comparison with paper-strip or free-solution electrophoresis. As many as 24 distinctly-separated components can be obtained from serum samples. Altho equally suitable for micro quantities, gel electrophoresis also accepts much larger quantities making it a practical method for preparative separations on a semi-micro scale; up to 200 mg. of protein mixture can be separated on one gel slab. If direct-contact cooling of the gel slab is employed, complete serum protein patterns can be obtained in less than two hours, showing better resolution than a 20-hour pattern on paper electrophoresis.

The gel material used as supporting medium may be hydrolyzed starch, agar, or the synthetic polyacrylamide Cyanogum (Reg. American Cyanamid Co.). Each of these media gives a different type of resolution, and in many problems it will be advantageous to employ all three concurrently or successively; the procedure then becomes analogous to two-dimensional chromatography. Migration thru a gel slab in the horizontal position introduces undesirable gravitational-convection effects in the migrating zone; therefore, a vertical gel slab is preferable, providing that a proper supporting apparatus is available.

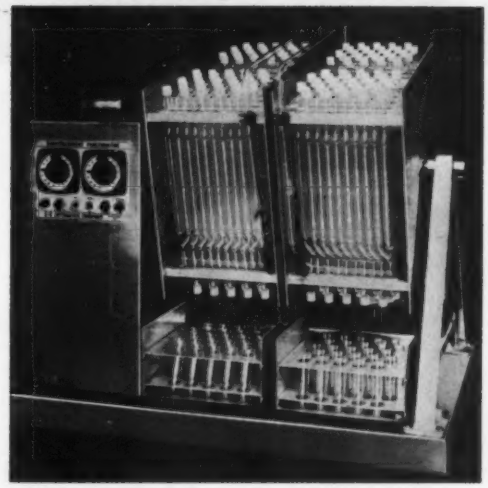
COUNTERCURRENT DISTRIBUTION

Counter-current extraction is a method of analyzing or separating the components of a mixture by dissolving it in one phase of a two-phase solvent system, and extracting it with the other phase. This effects a relative separation of the components according to the distribution co-efficients of each. Repeating the extraction many times in a systemic manner multiplies the effects of even small differences in distribution coefficients, so that closely similar substances can be separated. The separated volumes may be analyzed by any applicable method, preferably one which gives the total quantity of solute in each volume. Thus the total volume of solvent may be titrated when dealing with a mixture of acids. The most generally applicable, when non-volatile solutes are concerned, is the determination of the weight of solute in each fraction. It should be noted, however, that macro quantities of material are not required. Extremely low concentrations may be used, which may be far below those required for weight determinations. Necessary, of course, are sensitive analytical methods, such as color reactions or ultraviolet absorption. With such methods, counter-current extraction can analyze quantities as small as those in any other analysis. Results using low concentrations may be even more accurate than those with high concentrations owing to a closer approach to the laws of ideal solutions from which the following equations are derived. Counter-current extraction follows very exactly the Distribution Law

$$Ki = \frac{(i)\,1}{(i)\,2}$$

where K_i is a constant characteristic of the compound i; $(i)_1$, and $(i)_2$ are the concentrations of compound i at equilibrium in phase 1 and phase 2 of the solvent system. This fact allows a very mathematical analysis of the quantitative results obtained in counter-current extraction, and permits the direct comparison of experimental and theoretical results.

COUNTERCURRENT FRACTIONATOR



EC520 COUNTERCURRENT FRACTIONATOR with two racks of 100 20-ml tubes on base 24″ x 34″.

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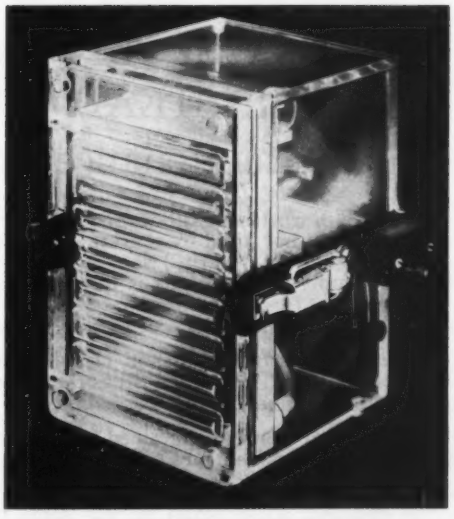
This apparatus will be demonstrated at Booth #101, National Chemical Exposition, Chicago, Sept. 5-8

VERTICAL GEL ELECTROPHORESIS



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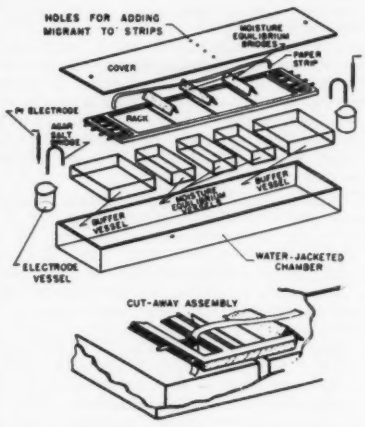

Figure 29. Details of the design of McDonald's ionograph for paper electrophoresis with free, horizontal strips.

The paper strips are thoroughly wetted by a buffer solution, and are mounted so that they rest lightly on the moisture equilibrium bridges, and are pulled taut by the spring-loaded end clamps. The ends of the paper strips dip into electrode compartments which are filled with buffer solution and which contain platinum electrodes for application of the electric field. Care is taken to insure that the paper strips are perfectly level and horizontal, and that the liquid levels in all compartments are exactly equal, so that there will be no tendency for liquid phase to siphon from one point to another. Since the passage of electric current through the liquid on the paper generates heat, it is also essential that heat be conducted away rapidly, so that it does not cause liquid to distill off the paper, thereby creating irregular flow patterns and interfering with the electrophoresis. In this instrument, the applied voltage and the consequent currents are moderate; for runs made at temperatures below room temperature, no special cooling is required. At room temperature, if the power input to the paper strip ($P = V \times I$) is 0.25 watts or more, the air in the chamber is replaced by helium, which has a much higher thermal conductivity.

Although it may be true in some cases that the electromobility of a component on a wetted paper strip is the same as it is in the free liquid, this is the exception rather than the rule. In general, adsorptive, ion exchange, and partition effects are of prime importance, and paper electrophoresis is a form of chromatography (as defined in this discussion) rather than of electrophoresis. This is true, whether or not solvent flow is per-

(*Continued on page A590*)

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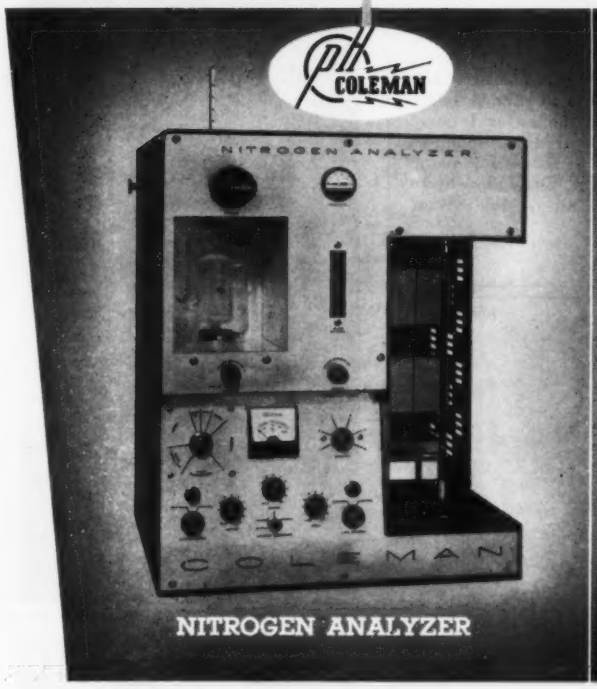
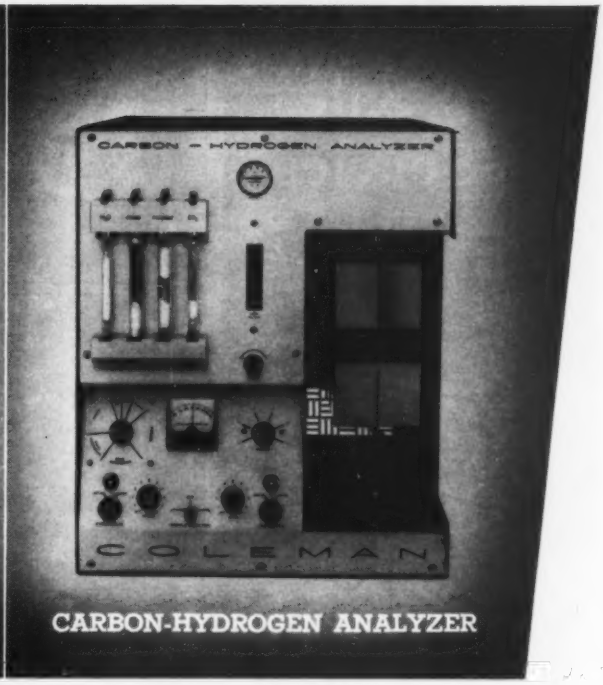


NITROGEN ANALYZER



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The Carbon-Hydrogen Analyzer performs a similar operation. It collects the combustion products of hydrogen (as water) and carbon (as carbon dioxide) in separate absorption tubes which are weighed after each analysis.

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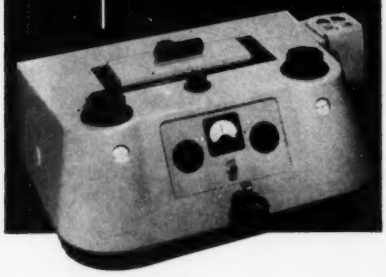

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mitted to contribute to the migration induced by the electric field.

In some paper electrophoresis devices, solvent flow is scrupulously avoided, as described above; in others, it is exploited to contribute to the selectivity and sensitivity of the chromatographic analysis. An example of the latter type of design is the Vertical Electrophoresis Tank (No. 2501, $100) of Shandon Scientific Co., Ltd., England, shown in diagram in Figure 30. In this device,

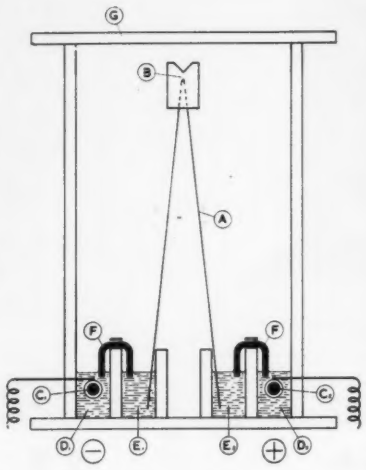

Figure 30. Design of the Shandon Vertical Electrophoresis Tank. *A.* Filter paper strip. *B.* Nylon string, or glass rod for support of paper. C_1 and C_2. Platinum coated electrodes. D_1 and D_2. Electrode buffer compartments. E_1 and E_2. Buffer compartments for the paper strips. *F.* Wicks connecting compartments *D* and *E*. *G.* Removable glass lid.

the paper strip is thoroughly wetted, and hung from its center over a nylon thread or glass rod, so that its ends dip into buffer reservoirs. The electrode reservoirs are separate, and communicate with the buffer compartments through wicks. This prevents the electrolysis products, formed as a consequence of the passage of current across the electrode/solution interface, from reaching the paper and altering the pH or other chemical conditions. When electric current is passing, there is considerable solvent flow up the strips, due both to electroendosmosis and to capillary action. The combination of these effects with the electromigration effect produces chromatographic separations that are in some cases (dependent upon the characteristics of the sample being analyzed) superior to those obtained with horizontal paper strips. This type of equipment is widely used for certain routine clinical tests.

Apparatus similar to the above is produced by Spinco Division, Beckman Instruments, Palo Alto, California (No. 63050, Durrum Type Electrophoresis Cell, $190).

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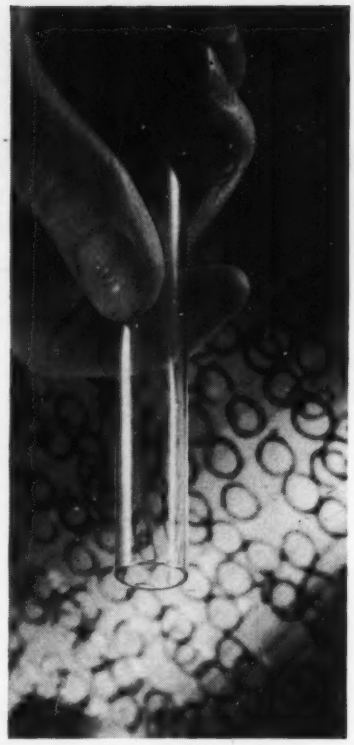

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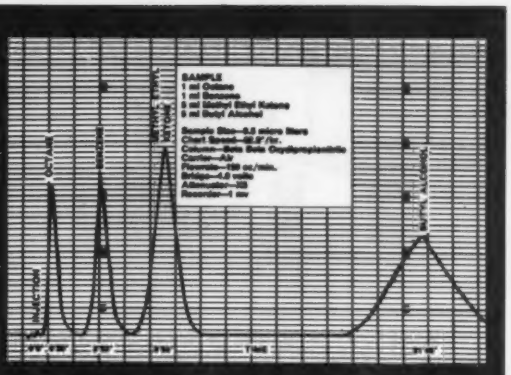

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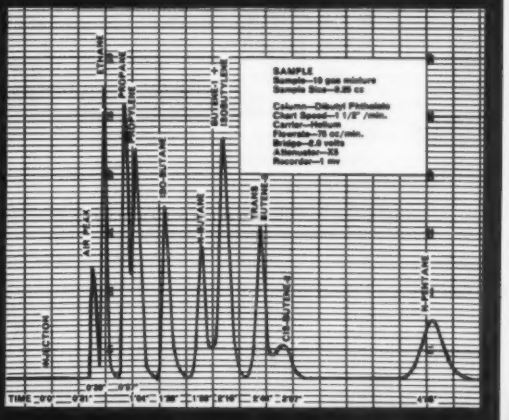

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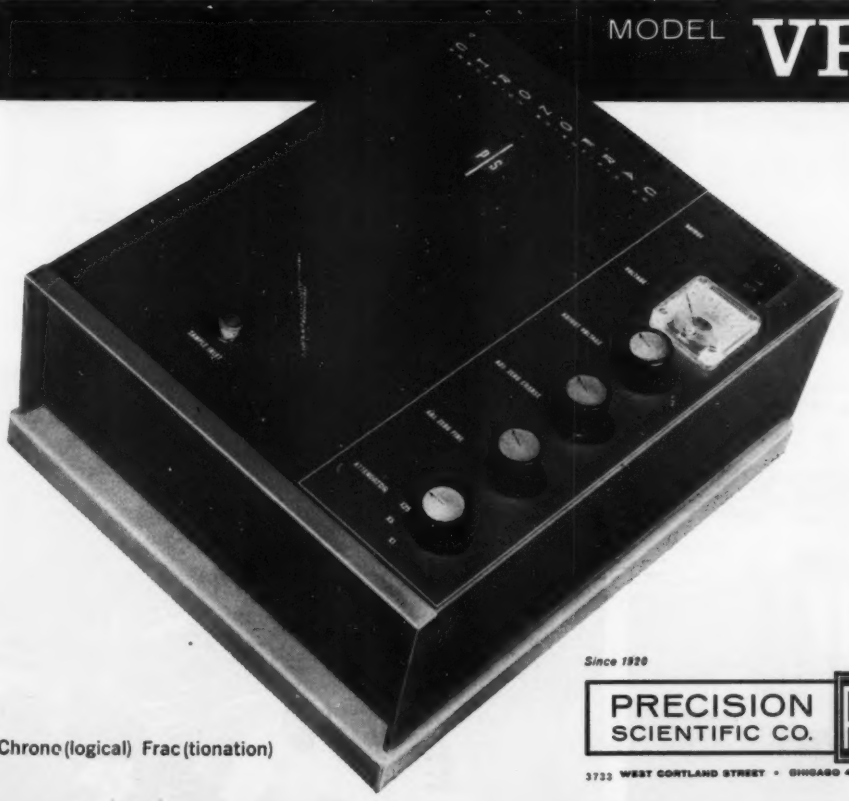

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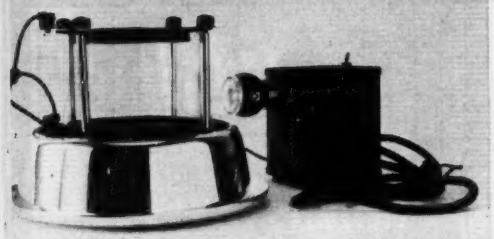

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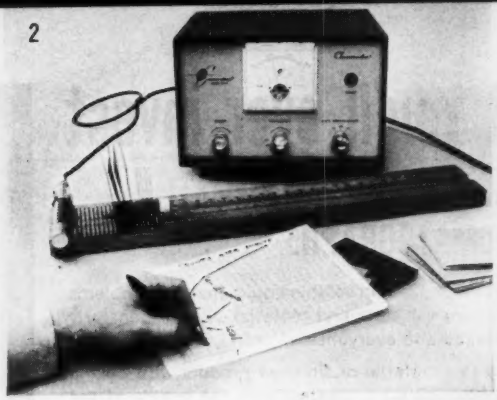

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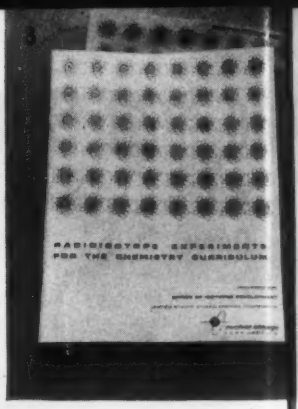

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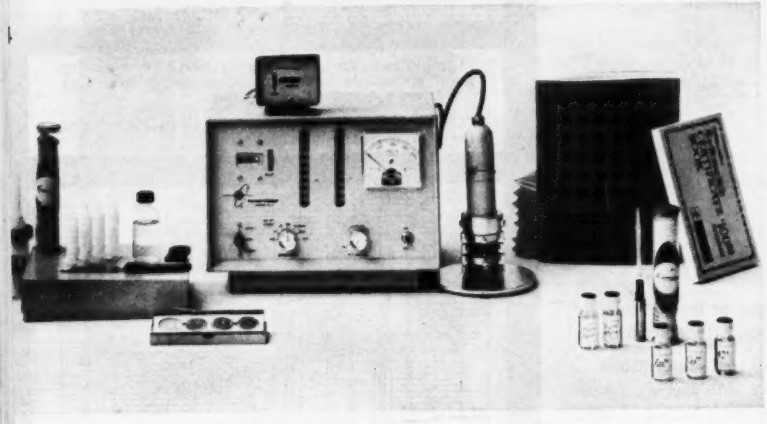

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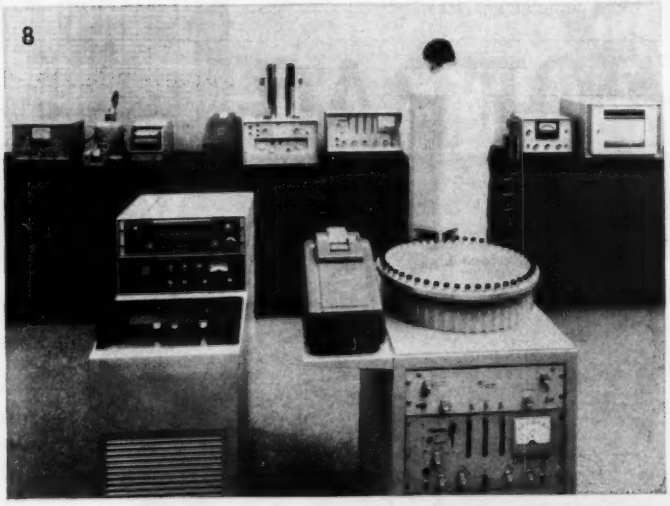

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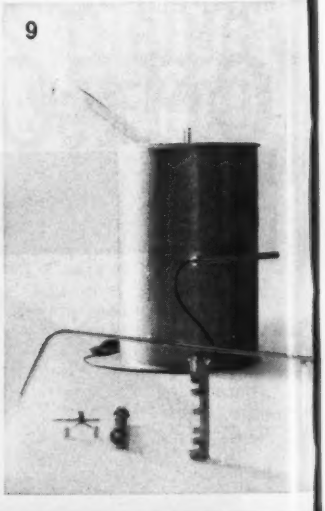

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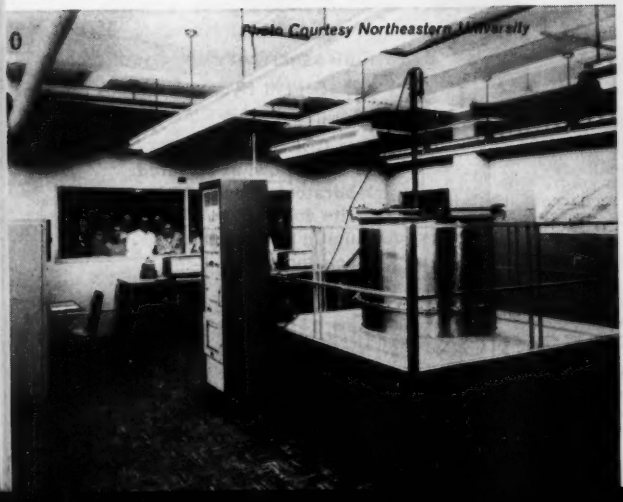

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11

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How to bring radioisotopes into your chemistry curriculum

Read how these Nuclear-Chicago products help to enlarge the scope of chemical education and also serve graduate and faculty research needs

RADIOISOTOPE procedures offer chemistry students penetrating insights into the laws of nature. Forward-looking educators are using the equipment illustrated at the left in lectures, for student experiments, and in research. These eleven photographs suggest the scope and completeness of Nuclear-Chicago's instrument line for teaching and research. Systems range from low-cost equipment for demonstration to sophisticated instrumentation for fundamental investigation. All are characterized by reliability, precision, versatility, and inherent safety.

Chemistry departments now employing radioisotope techniques will want to consider program expansion to take advantage of the newer products illustrated. Others may well regard the apparatus presented here as a planning guide.

Low-cost demonstration equipment for the nuclear age became a reality with the introduction of Nuclear-Chicago's Cloudmaster® and Classmaster®. The Cloudmaster (figure 1) is a continuously sensitive cloud chamber that provides a spectacular display of ionization tracks caused by alpha, beta, gamma, and meson radiation. The Classmaster (figure 2) is a complete system for demonstrating radiation absorption, scattering, geometry effects, coincidence loss, etc. In addition, it permits simple tracer experiments using license-exempt radioisotopes. Both instruments enjoy wide acceptance because of their simplicity of operation and the vivid, easily-understood presentations they make possible.

Thirty-three student experiments that fit naturally into the chemistry curriculum without requiring changes in basic course content are contained in a new 187-page experiment manual and 60-page companion volume of instructor notes (figure 3) developed by Nuclear-Chicago under contract with the Atomic Energy Commission. Model 4001 nuclear training system (figure 4) is offered with isotopes, sample preparation equipment, and instruments to perform all the experiments covered in the manual. The system features ease of sample preparation using the chimney funnel shown in figure 5.

Model 4001 is supplied with the long-lived radionuclide set RNS-110 (figure 6) and the radionuclide certificate book RCB-1 (figure 7) with which the instructor can order short- or long-lived isotopes as they are needed. Neither requires an AEC license to purchase or use, and both are available individually.

As student mastery of radioisotope procedures advances, a chemistry department will next want to consider establishing a complete nuclear laboratory for precision handling, detection, and counting of beta- and gamma-emitting isotopes. Such a facility has valuable application to qualitative and quantitative studies at both the graduate and undergraduate levels. A typical installation is pictured in figure 8. In the left foreground is the Model 701 liquid scintillation detector with Logic scaler, and at the right is the Model C120 automatic sample changer for gamma-emitting liquids or solids in test tubes. In the background, from the left, are the Actigraph II® system for scanning radiochromatograms, a low-background automatic sample changer for solid beta samples, and the Dynacon® electrometer for high-efficiency measurement of beta samples in the gaseous phase.

At this stage, the ideally equipped university will lack only the means to create its own radioactive isotopes and to study reactor techniques and fundamental nuclear properties. For these basic needs, Nuclear-Chicago offers a three-step program.

First, undergraduate departments will choose the NH3 neutron howitzer (figure 9) for experiments with low-level neutron sources, production of short-lived, low-intensity radioisotopic tracers, and low-flux activation analysis.

Second, the chemistry, physics, and engineering faculties will often cooperate in establishing an advanced training facility centered on the inherently safe Model 9000 subcritical assembly (figure 10). Characteristics of this water-moderated, natural uranium assembly are nearly identical in nature to those of a full-scale power or research reactor, and the student readily masters the fundamentals of reactor theory and design.

Finally, the long-felt need of university research scientists for a versatile, relatively inexpensive, high-intensity neutron source can be well satisfied by the TNC neutron generator designed and manufactured by Nuclear-Chicago's subsidiary, Texas Nuclear Corporation (figure 11). Providing a controllable yield of 4×10^{10} neutrons per second and a thermal neutron flux of greater than 10^8 using an appropriate moderator, the generator is widely employed in activation analysis, process control, applied research and development, and fundamental investigation.

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Chemical Instrumentation

A convenient arrangement for horizontal free paper electrophoresis is the Shandon Universal Electrophoresis Apparatus (No. 2548, $138) shown in Figure 31.

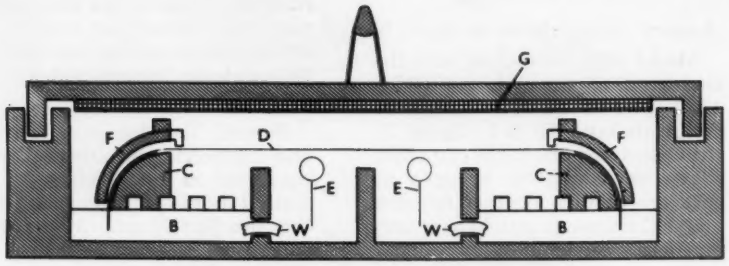

Figure 31. Shandon Universal Electrophoresis Apparatus. *B.* Buffer compartments. *C.* Paper strip supports. *D.* Paper strip. *E.* Electrodes. *F.* Paper holders. *G.* Sponge pad. *W.* Wicks.

The special features of this design are the large buffer compartments, which help to maintain temperature uniformity; the adjustable positioning of the paper holders, to accommodate strips from 12 to 18 cm in length; and the sponge pad on the underside of the cover, to soak up condensation liquid and aid in vapor saturation.

A more elaborate apparatus designed around the free horizontal suspension of paper strips is the Zone Electrophoresis Apparatus available from Buchler Instruments, New York 31, N. Y. (No. 3-1000, complete with power supply, $382), illustrated in Figure 32. This unit has a safety interlock that disconnects the power whenever the cover is up; a tension bar rests on the paper strips and takes up any slack that may develop during a run. The paper support can be removed as a unit from the electrophoresis chamber and placed in an oven for drying of the strips.

An instrument for supported horizontal paper electrophoresis is the Paper Electrophoresis Migration Chamber manufactured by Research Specialties Co., Richmond, California (Model 1400, $255). The paper sheet or strips rest on a water-cooled table, so that temperature uniformity and constancy can be precisely maintained over the entire migration path. This permits relatively high voltages to be employed for the electrophoresis without experiencing difficulties due to evaporation or thermal instability of sample constituents. The unit may also be used with solid media such as starch gel by depositing the latter within end gates placed on a wetted paper sheet which serves as wicks and forms a conducting path between the electrodes and the solid medium. A schematic diagram of this arrangement is given in Figure 33.

Other manufacturers of paper electrophoresis instruments include:

Microchemical Specialties Co., Berkeley 10, California—*a supported horizontal paper chamber, and a multi-cell chamber.*

E-C Apparatus Co., Swarthmore, Pennsylvania—*a supported horizontal paper unit with Teflon migration surfaces.*

Matthew Laboratories, Yonkers 2, New York—*free horizontal paper chambers.*

LKB Instruments, Inc., Washington 14, D. C.—*horizontal paper rests on embossed points of a plastic cassette.*

Bender and Hobein, Germany, distributed in U. S. by Harshaw Scientific Co., Cleveland 6, Ohio—*free horizontal paper chamber.*

Servonuclear Corp., Long Island City 2, N. Y.—*high voltage paper electrophoresis units.*

Bibliography

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(*Continued on page A600*)

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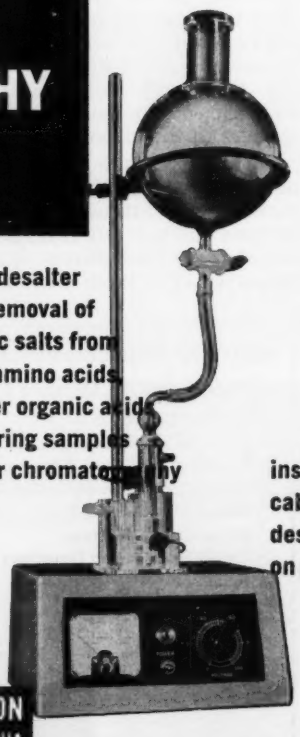

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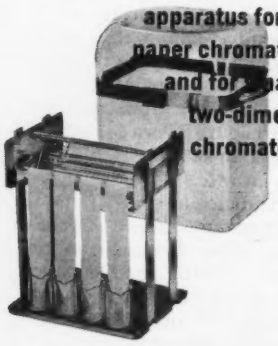

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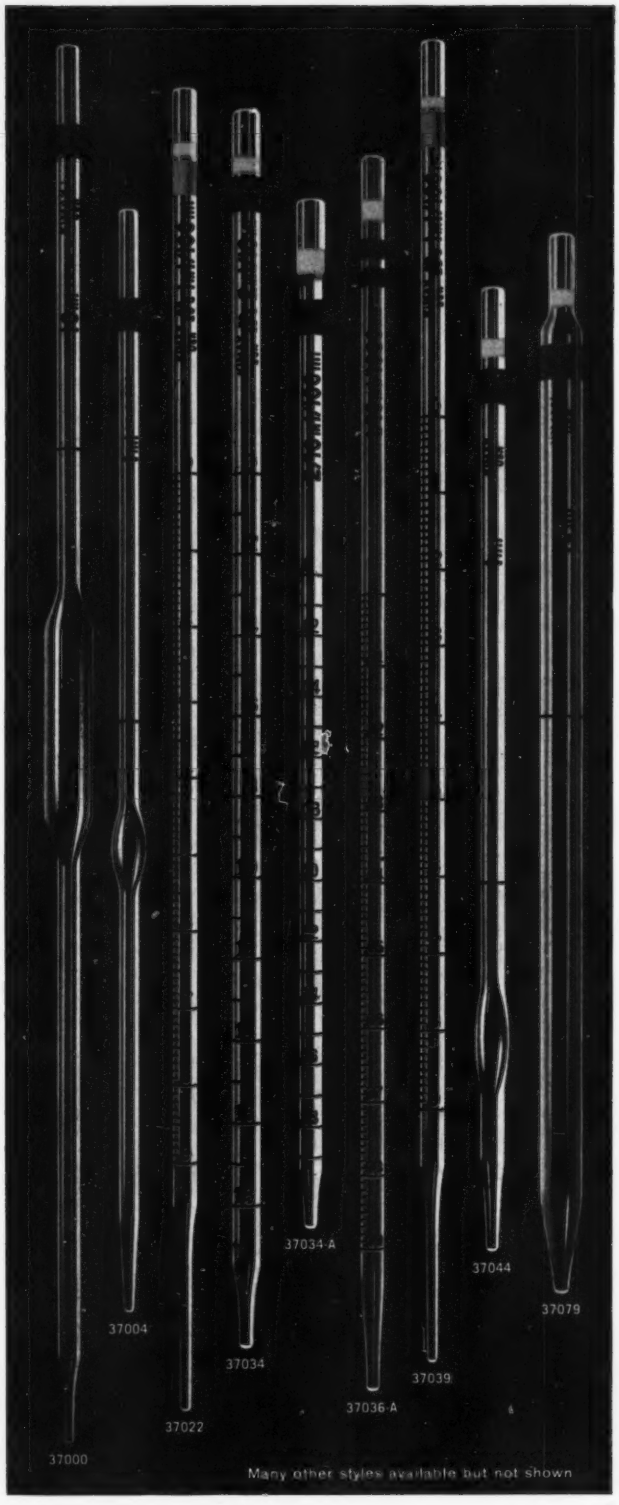

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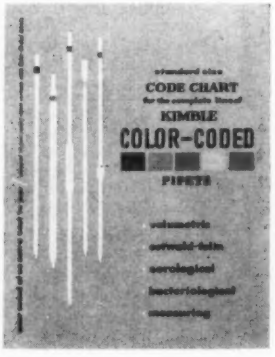

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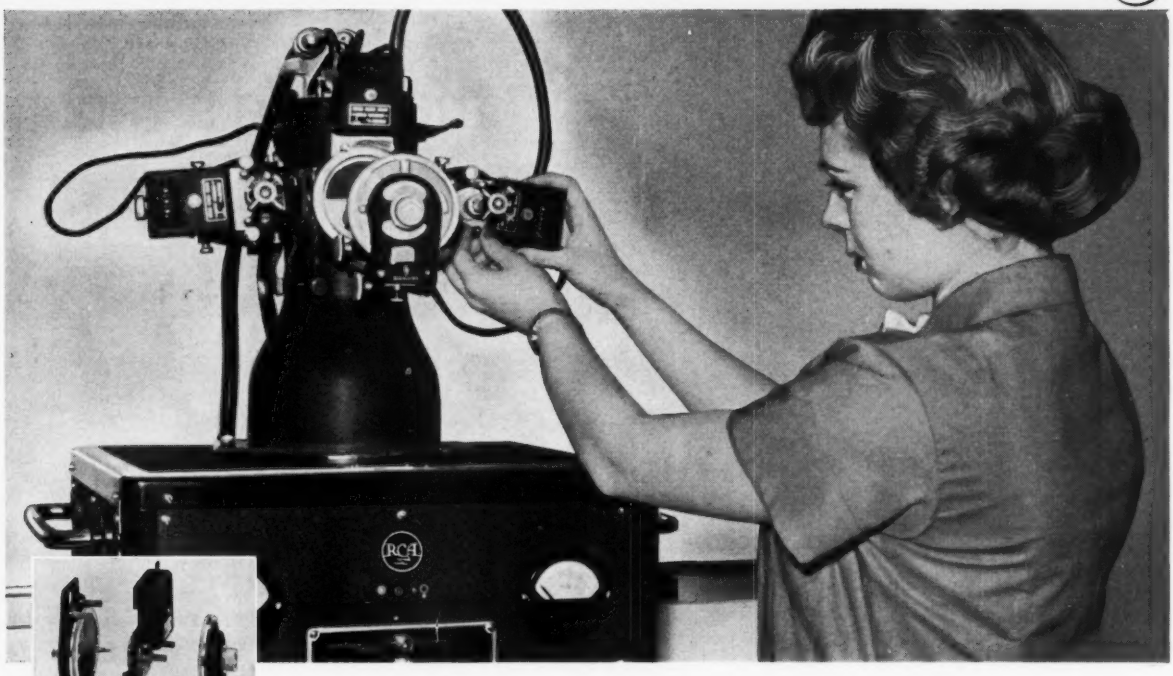

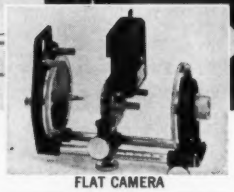

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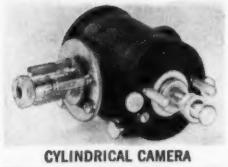

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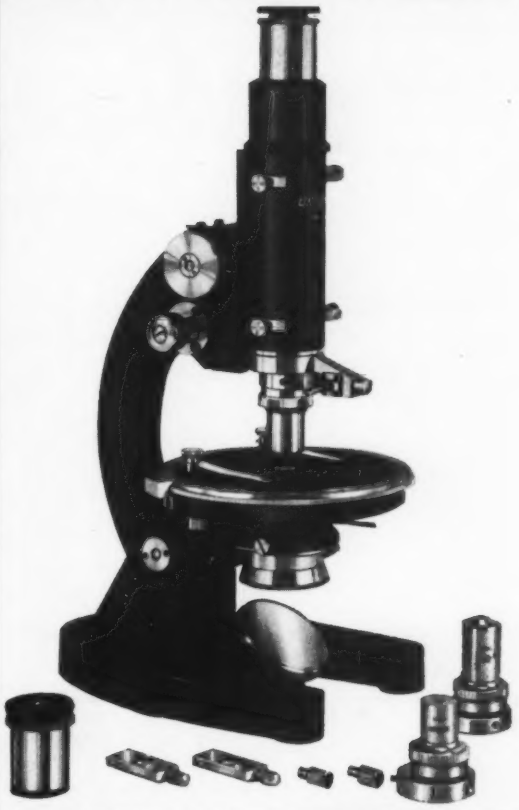

1 Iris diaphragm
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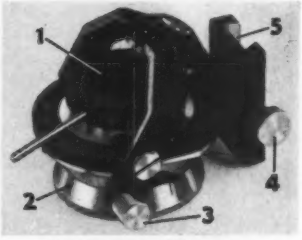
3 Swing-out lever
4 Focusing lock
5 Focusing slide



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ZWEIG, G., "Paper Chromatography. A Laboratory Manual," Academic Press, N. Y., 1952.

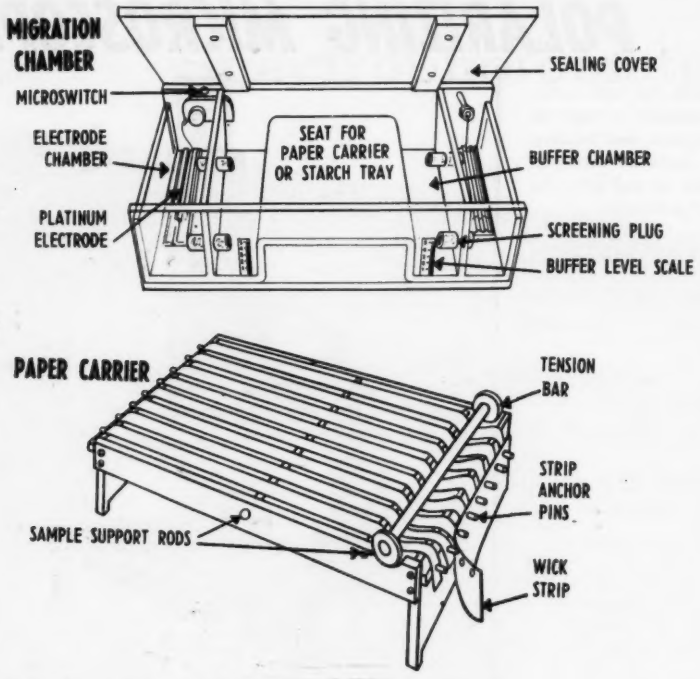

Figure 32. Design of the Buchler Zone Electrophoresis Apparatus.

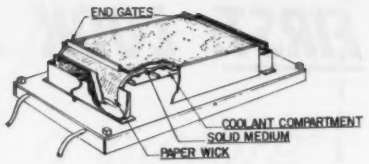

Figure 33. Diagram of the Research Specialites Co. paper electrophoresis apparatus as used for work with starch gel or other solid media. Only the water-cooled table and its supporting base plate with the buffer compartments are shown.

CASSIDY, H. G., "Fundamentals of Chromatography," in "Technique of Organic Chemistry," A. Weissberger, ed., Interscience, N. Y., vol. 10, 1957.

CHRISTIAN, S. D., "Laboratory Countercurrent Distribution Apparatus," J. CHEM. ED., 36, 195 (1959).

HAGDAHL, L., "On the Use of Coupled Columns in Chromatography," Science Tools, 1, 21 (1954); available from LKB Instruments, Inc., 4840 Rugby Avenue, Washington 14, D. C.

KARLER, A., "Topics in Electrochromatography. Curtain Continuous Flow Electrophoresis," 1958. Available from Karler Laboratories, El Cerrito, California.

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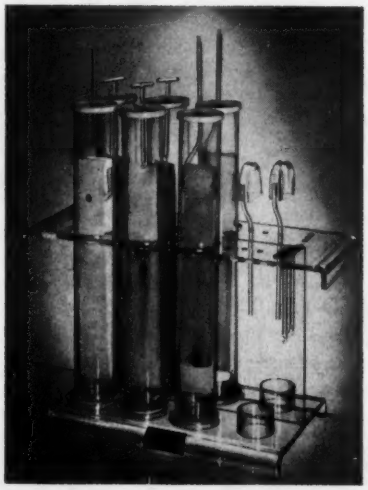

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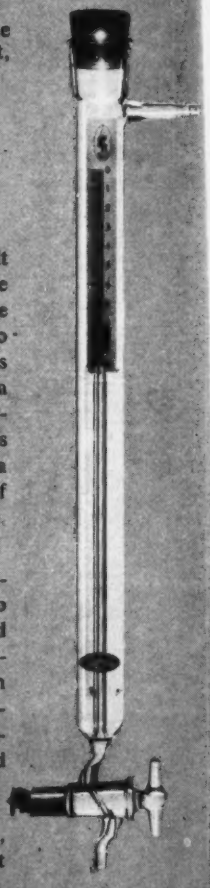

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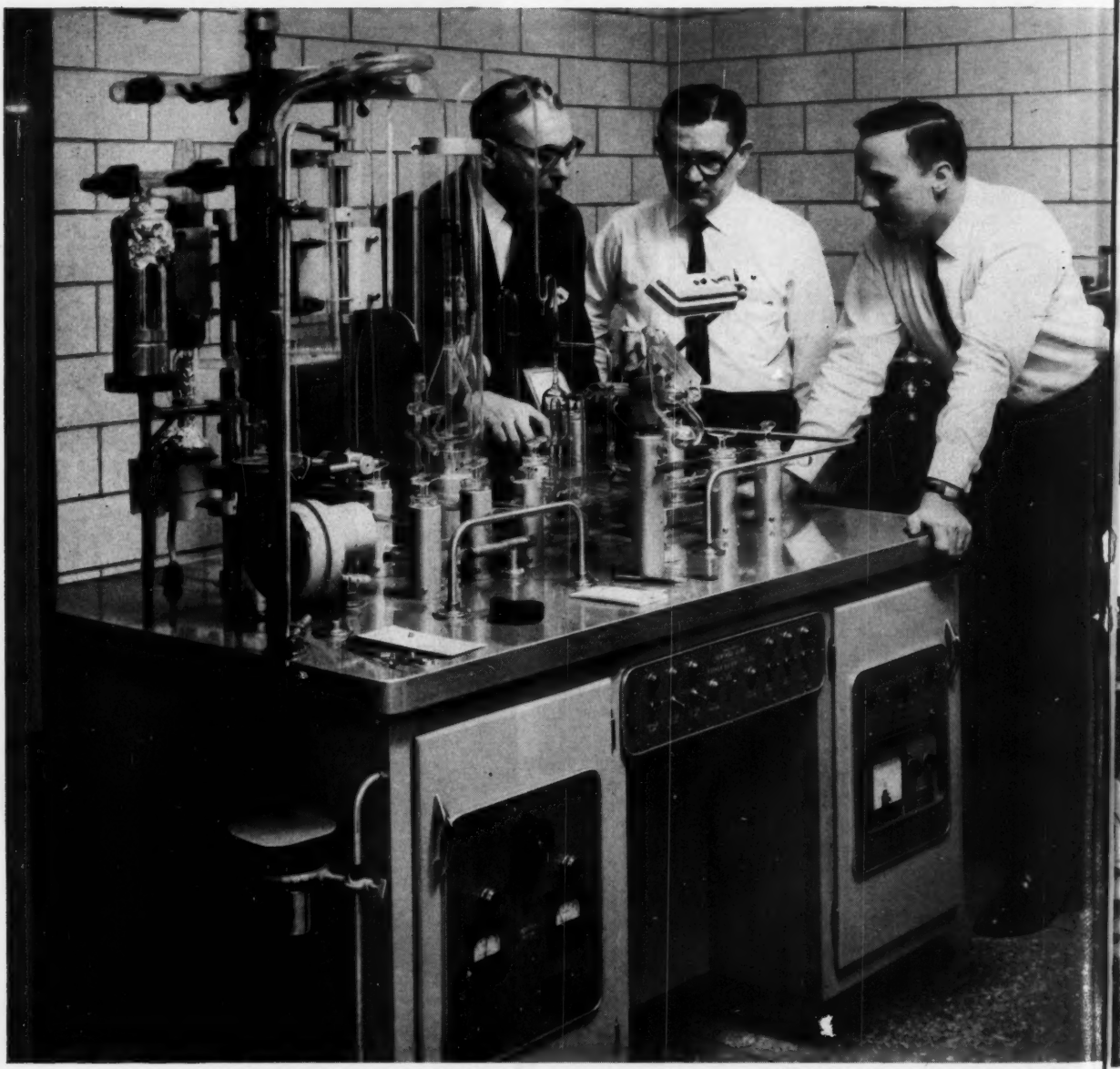

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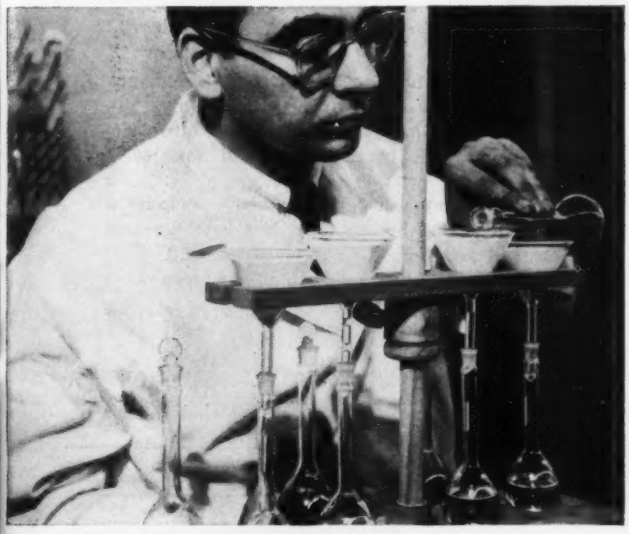

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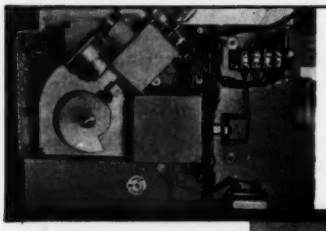

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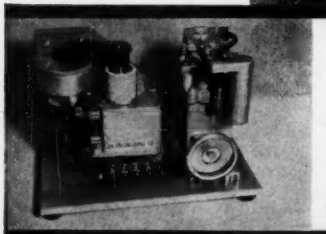

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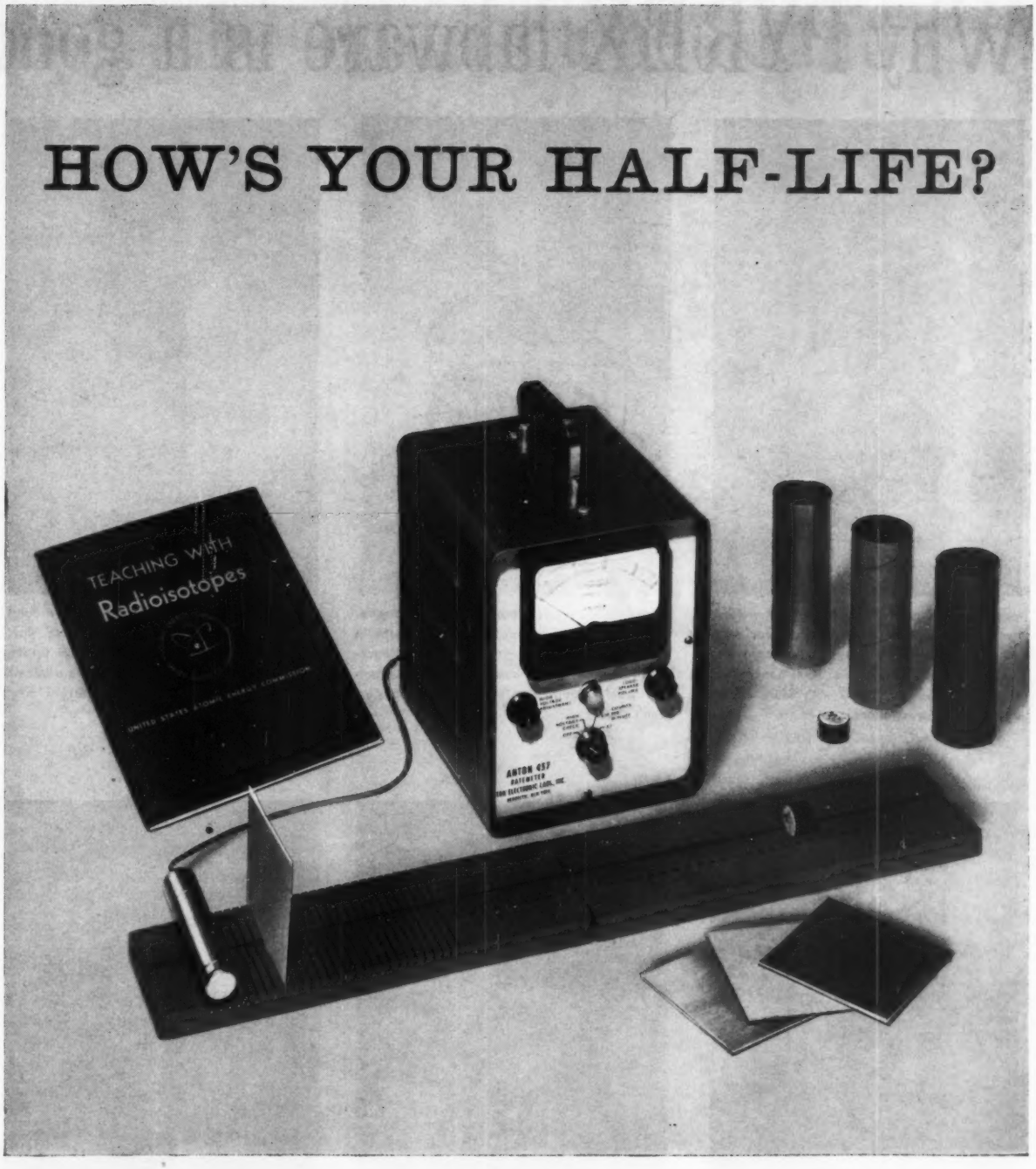

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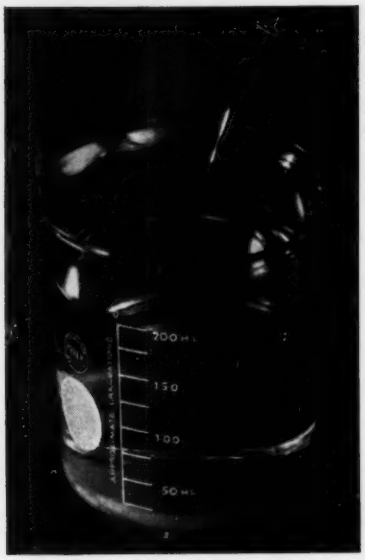

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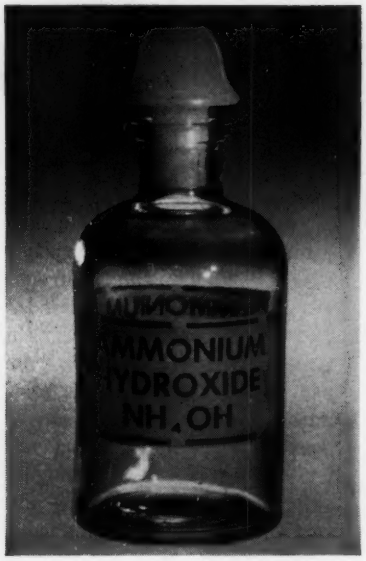

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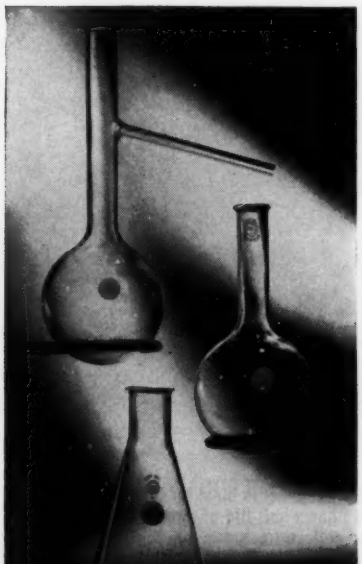

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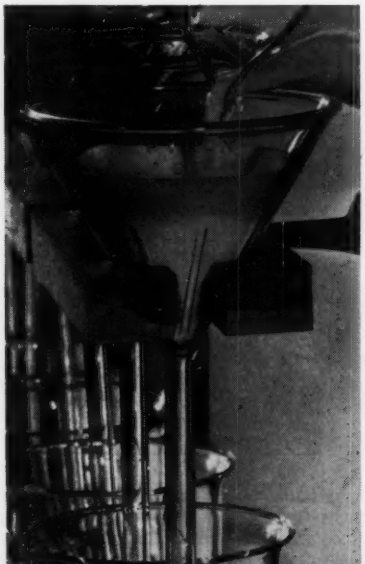

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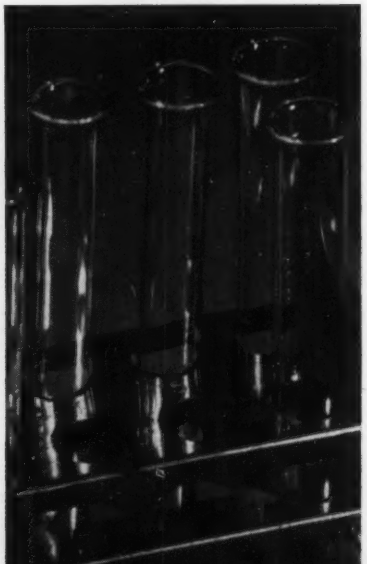

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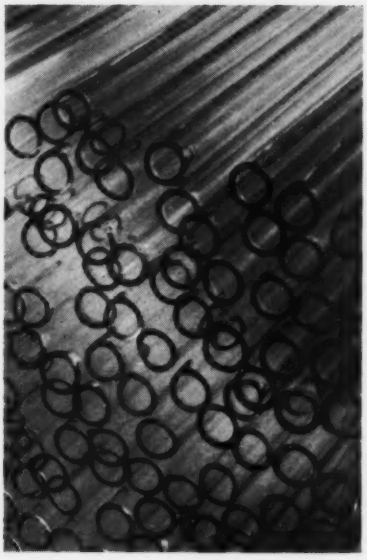

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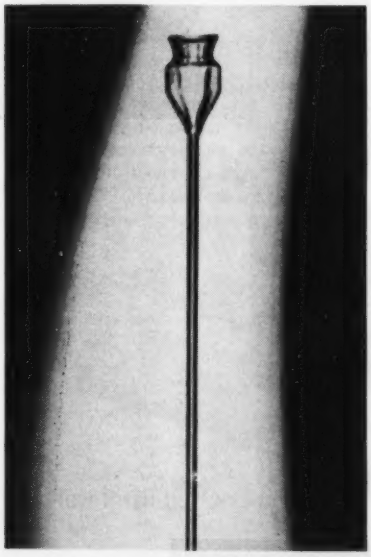

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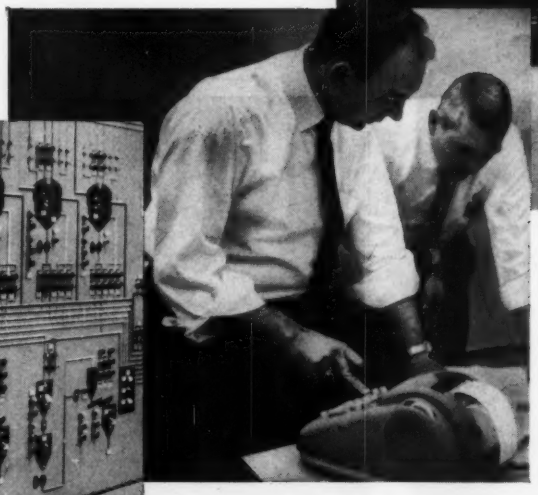

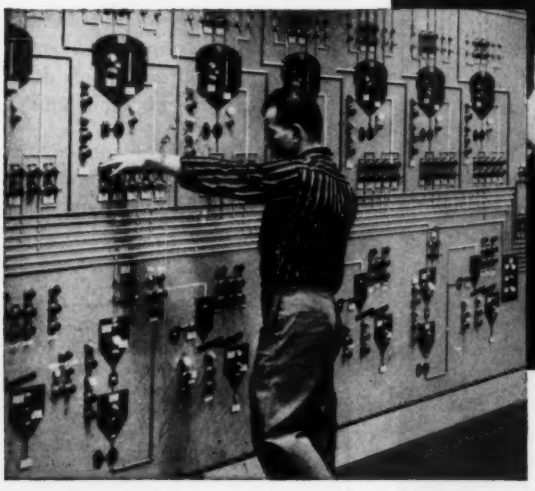

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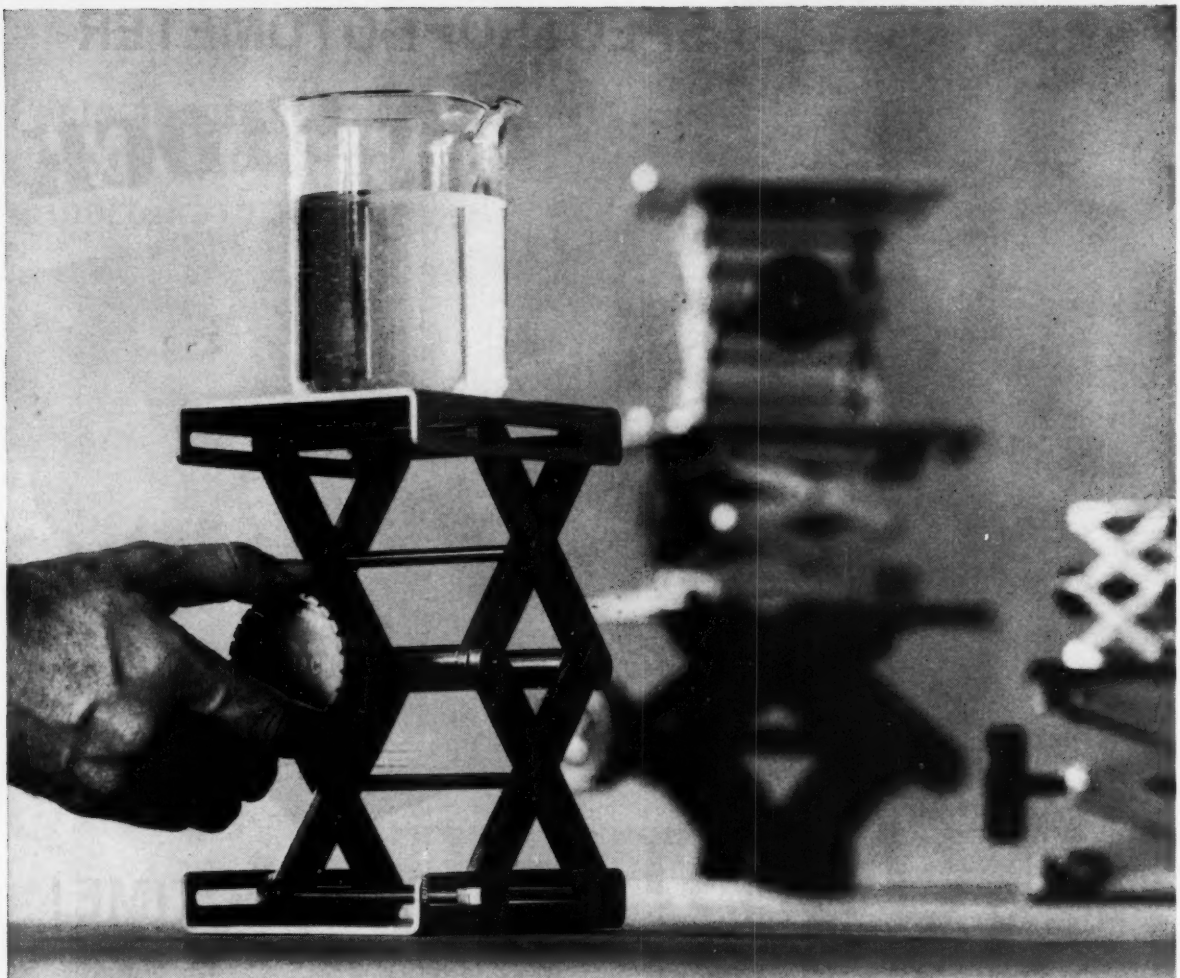

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Compiled by **JAY A. YOUNG,** Kings College, Wilkes-Barre, Penna.
JOHN K. TAYLOR, National Bureau of Standards, Washington, D. C.

Demonstration of the Chemistry of the Permanent Wave

See ROBINSON, T., AND DANIEL, L. J., *J. Chem. Educ.*, **33**, 583–4 (1956).

The resiliency of human hair can be attributed to the disulfide bonds that stabilize the folded peptide chains which comprise the major portion of this fiber. If a large percentage of these disulfide bonds are reduced to sulfhydryl groups the hair becomes flaccid. Then, if the hair is bent or otherwise distorted, and the sulfhydryl groups oxidized, new disulfide bonds will be formed between pairs of sulfur atoms that were not previously joined to each other, and the hair will tend to retain its new shape, "locked," in position by the new disulfide bonds.

Robinson and Daniel describe an interesting, simple experiment that illustrates the preceding statements, using commercially available home permanent wave kits for this demonstration.

Questions:

What denaturing (reducing) agents, oxidizing agents, and catalysts would be suitable for imparting a permanent wave to human hair formed into a wig? (That is, you need not consider any possible adverse affects of the compounds selected on the skin of the scalp.) Test your ideas using samples of hair, and devise a quantitative method for measuring the "permanence" of the curls you make.

Can you devise a process for forming a permanent crease, or press, in wool cloth?

Are there any synthetic fibers which can be permanently distorted by chemically breaking and subsequently reforming bonds that form cross links between adjacent sub-molecular units?

Inhibition of Carbonic Anhydrase by Thiocyanate

See MCGEACHIN, R. L., *J. Chem. Educ.*, **32**, 191–2 (1955).

Carbonic anhydrase, an enzyme which is present in animal (including human) blood, catalyzes the decomposition of carbonic acid to carbon dioxide and water. Like other enzymes, it is inhibited, to a greater or lesser degree, by many substances. For example, in the presence of thiocyanate ion, the catalytic action of carbonic anhydrase is reduced, and this inhibition increases as the concentration of thiocyanate ion is increased.

McGeachin has studied the effect of thiocyanate ion on the rate of the reaction

$$CO_2 + H_2O \rightarrow H_2CO_3$$

which is also catalyzed by carbonic anhydrase. The reaction was followed by noting the time required for bromthymol blue to change color when CO_2 was bubbled at a reproducible rate into a mixture of aqueous buffer, blood, bromthymol blue, and thiocyanate ion in varying concentrations.

Questions:

Can satisfactory determinations of the inhibitory effect of thiocyanate ion on carbonic anhydrase be obtained by modifying McGeachin's procedure, using a small piece of dry ice (about 0.5 ml volume) inserted directly into the aqueous mixture, instead of using CO_2 from a tank of compressed gas or a Kipp generator?

What other substances can you find which inhibit the catalytic action of carbonic anhydrase? Compare their effectiveness quantitatively if possible, with thiocyanate ion.

Is the quantity of carbonic anhydrase, present in equal volumes, the same for many different kinds of animal blood?

Does the concentration of carbonic anhydrase present in your blood vary with the vigor of muscular activity, just prior to extracting the sample of blood to be examined? If so, suggest an explanation and test by further laboratory work.

Is carbonic anhydrase present in a chloroform extract of red blood cells?

Model Glucostat

See LOWE, H. J., *J. Chem. Educ.*, **32**, 304–6 (1955).

In an interconnected assembly consisting of a series of flasks, tubes, valves, and other equipment, the movement of liquid through these vessels is manually manipulated to illustrate the mechanisms by which glucose is transferred across cell membranes against a concentration gradient. Intestinal absorption of glucose, postprandial hyperglycemia, the role of adenosine triphosphate, hyperinsulinism, hypoadrenalism, glycosuria, and other similar conditions can be simulated in the apparatus.

Questions:

Although the construction and consequent study of the operation of this model would be valuable for any interested student, such a project would not qualify as an example of the kind of work this series has encouraged, because no laboratory investigation is necessary. However, the construction of this model could serve as the starting point for a laboratory investigation. For example, does the model accurately simulate the phenomena associated with the intestinal absorption of glucose, with hyperinsulinism, or other condition as determined from a study of these *in vivo* or *in vitro*?

Can you construct a similar model, illustrating a different biochemical system, from your studies of that system in laboratory animals?

Show, by means of a model, based on your laboratory investigations, the phenomena associated with a selected series of biochemical reactions that occur in a living plant.

EDTA and Complex Formation

See JOHNSTON, M. B., ET AL., *J. Chem. Educ.*, **35**, 601–6 (1958).

Chelon Approach to Analysis (I) and (II)

See REILLEY, C. N., ET AL., *J. Chem. Educ.*, **36**, 555–64 and 619–26 (1959).

Ethylenediaminetetraacetic acid and its anion form, ethylenediaminetetraacetate (EDTA is used as an abbreviation for either form) is a hexa-dentate ligand which forms water soluble complexes with many different metal ions, usually in a 1:1 mole ratio. Usually, the complex with a metal ion can be formed in one step, by addition of a solution of the sodium salt of EDTA to a solution containing the metal ion. Various indicators are available which, by their color, indicate the presence or absence of free metal ion. The stability of the complex that is formed depends upon the metal ion and upon other solutes that are also present in the solution. Hence by varying these conditions, and by using a proper indicator, many different metals can be titrated with a solution of the sodium salt of EDTA, and the quantity of metal ion can be determined by direct or by indirect means.

Within the past few years, the versatility of EDTA and other chelons as complexing agents and as quantitative analysis reagents has been studied by many investigators; almost daily, new and significant uses are being found for these remarkable reagents. The three articles cited above discuss the properties of chelons, emphasizing EDTA, and suggest more than 40 different experiments which illustrate these properties and their analytical applications in detail. For a related general discussion, see MARTELL, A. E., *J. Chem. Educ.*, **29**, 270 (1952).

Additional published information is available upon request from three industrial organizations, those mentioned in references (*12*), (*13*), and (*14*) in Johnston, *et al.*; Dr. Barnard (one of the co-authors) has specifically indicated his willingness to help students who write to him requesting copies of the literature available from his company. However, any such requests should be specific, courteous, and sincere. Before requesting assistance, the student should read the editorial on this topic in J. CHEM. EDUC., **38**, 159 (1961) and follow the recommendations given there.

Since several questions are stated in the three articles and many others can be inferred by the reader, none need be appended here.

The Standardization of Sodium Hydroxide Solution

See FEHLANDT, P. R., *J. Chem. Educ.*, **26**, 322–3 (1949).

The concentration of a sodium hydroxide solution can be determined by delivering a known volume, measured in a graduated cylinder, into an evaporating dish where it is neutralized with a slight excess of hydrochloric acid. An indicator is used to determine when an excess of acid has been added. The solution is carefully evaporated to dryness and the sodium chloride residue is weighed.

Questions:

Within the limits of precision obtainable when using a graduated cylinder and the most precise balance available in your laboratory, does it make any difference whether you use a soluble indicator, such as phenolphthalein or methyl orange, or whether you use a piece of indicator paper, such as litmus paper, and wash any solute that has accumulated in the paper back into the aqueous solution, using distilled water?

Weigh any suitable stable object several times on your laboratory balance, to establish the limits of precision of your weighing technique. Then, determine the concentration of sodium hydroxide in several aliquots of a well mixed solution. Assuming that all additional error is due to lack of precision in the reading of the meniscus level in your graduate, determine the standard deviation of your readings of the meniscus level of your graduate.

Show that a pipet is a more precise volume measuring device than a graduate.

In order to obtain a better measure of the precision of your readings of the meniscus level of your graduate (or pipet), it would be desirable to use hydrobromic acid or hydroiodic acid instead of hydrochloric acid. Show that this statement is true.

The pellets or sticks of sodium hydroxide in the reagent bottle in your laboratory will react with carbon dioxide in the air, every time the bottle is opened. As a result, the sodium hydroxide in the bottle is contaminated with sodium carbonate and sodium hydrogen carbonate. The procedure described by Fehlandt will not disclose that a solution of sodium hydroxide prepared from such contaminated material is, indeed, impure. Can you modify Fehlandt's procedure to determine the per cent purity of solid sodium hydroxide and the percentage of the two major contaminants, sodium carbonate and sodium hydrogen carbonate?

Experiments with Ash

See FROMM, F., *J. Chem. Educ.*, **24**, 231 (1947).

Potassium carbonate can be extracted from wood, charcoal, or tobacco ash. A procedure for the approximate determination of the quantity of potassium carbonate in a 0.1–0.2 gram sample of ash is given by Fromm, as well as a procedure for the extraction of this substance from a larger sample of ash and for the preparation of "potash soap."

Questions:

The procedure for estimating the potassium carbonate content of ash is given, without explanation, in the article. Show that this procedure is, or is not, suitable for the estimation of the potassium carbonate content of an ash by determining the reactions that occur when the procedure is followed.

Can you devise a procedure for determining potassium carbonate in ash which will yield a more accurate result?

Ash is a heterogeneous material; hence, two different samples of ash, each weighing less than one gram, taken from a quantity of ash weighing several grams, might be expected to show marked differences in percentage composition. Devise a sampling procedure which will minimize the variation found in the composition of two or more different small samples.

What other constituents of wood (charcoal) ash can you identify and measure quantitatively?

Tobacco ash has a much higher percentage of potassium carbonate than charcoal ash, according to Fromm. What other compositional differences in these two kinds of ash can you establish?

Is it possible to identify different varieties of wood by quantitative analysis of the corresponding ashes for one, or perhaps two, constituents?

Coal ash is quite different in appearance from wood ash. How different is it in composition?

Is there a difference between the ashes formed from bituminous and anthracite coal? (*Note:* Students who undertake a study of this question and who find anthracite coal difficult to obtain may apply to one of us, J.A.Y., for a 50-gram sample of anthracite.)

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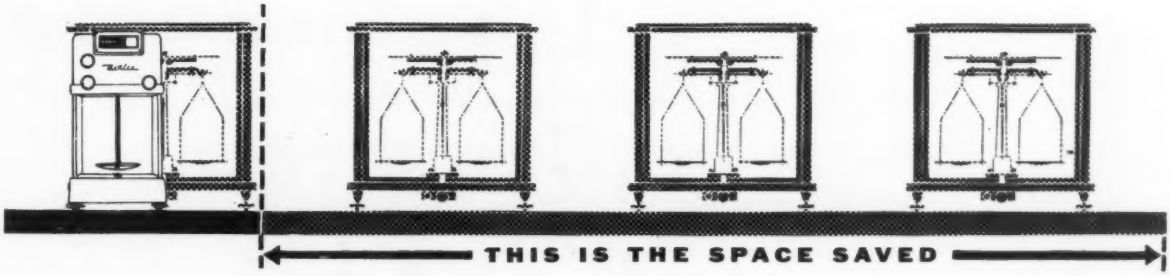

The diagram is based on the conservative assumption that one Mettler balance does the work of four equal-arm, undamped student balances.

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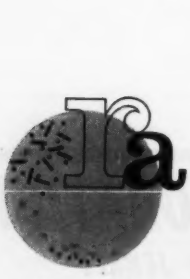

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Chem Ed

Out of the EDITOR'S BASKET

Edited by William G. Kessel

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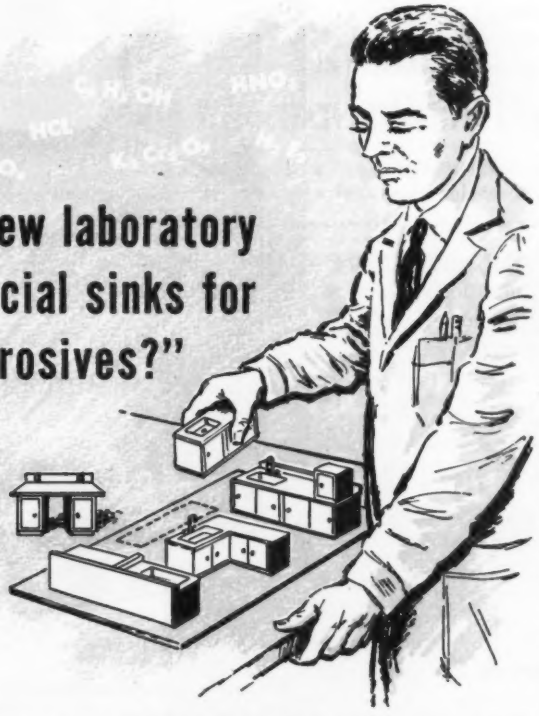
New Apparatus and Equipment

▶ Roger Gilmont Instruments, Inc., 1 Great Neck Rd., Great Neck, New York, announces the development of a new concept in flow measurement—calibrated and correlated flowmeters. Based on Stokes' law, the new correlation permits the calculation of a calibration curve for any fluid whose density and viscosity are known.



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▶ "Chirpee" a new fountain-pen-size continuous radiation monitor, that chirps and flashes when its wearer encounters a radiation field, is now available from Atomic Accessories Inc., 811 W. Merrick Rd., Valley Stream, New York.

▶ A new nitrogen generator, produced by Joy Manufacturing Co., Henry Oliver Bldg., Pittsburgh 22, Pennsylvania, ensures laboratory and industrial users continuous production of liquid nitrogen at a guaranteed rate of 20 pounds an hour. Low speed reciprocating components in the Joy LN-20 eliminate the need for a special foundation, and ensure low noise levels.

▶ Research in solid propellants at the Redstone Arsenal, Huntsville, Alabama demands strictly controlled atmospheres for handling extremely hygroscopic and moderately oxygen-sensitive materials. To conserve lab space and reduce equipment costs, Rohm & Haas Company's Redstone Arsenal Research Division at Huntsville is now using inexpensive flexible glove cabinets supplied by the Torsion Balance Co., Clifton, New Jersey.

▶ Kol, Inc., 2323 Ellis Ave., St. Paul 14, Minnesota, announces the addition of a new all-purpose batch mixer for industrial use to their line of portable mixers. The M-61 Minal is especially designed for factory, laboratory, demonstration and school uses. It can mix chemicals, plastics, cements, adhesives, paints, foods, inks, epoxy, latex, concrete and tar.

▶ Edmund Scientific Co., Barrington 54, New Jersey offers a new graph rubber stamp kit including a 100 block per inch, a 16 block per inch and a polar coordinate graph stamp with 3 in. diameter with 5° markings.

▶ Write Delta Design, Inc., 3163 Adams Ave., San Diego 16, California, for information on their temperature monitor. This device can be used to limit temperature between two pre-set limits; when the maximum or minimum is reached, the device shuts off and must be re-set manually.

▶ Three new, low-cost, portable potentiometers have been announced by Leeds & Northrup Co. These instruments provide on the spot measurements of temperature for instrument maintenance departments, and in testing and research work. For more information, write Leeds & Northrup Co., 4934 Stenton Ave., Philadelphia 44, Pennsylvania, for *Data Sheet E-33(5)*.

▶ Nuclear-Chicago Corp., 359 E. Howard Ave., Des Plaines, Illinois, has developed a high range Cutie Pie survey meter for rate of dose and accumulative dose radiation measurements. The battery-operated instrument, designated the Model 2586-T, is supplied with two interchangeable ionization chambers. A rate of dose chamber has ranges of 0–2.5, 0–25, and 0–250 reontgens per hour. An accumulated dose chamber has ranges of 0–0.25 0–2.5, and 0–25 roentgens.

▶ Bausch & Lomb, Inc., Rochester 2, New

(*Continued on page A617*)

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York, has announced a series of newly designed standard metallurgical microscopes at prices lower than any current domestic instruments of this type. The STM provides vocational and engineering schools, colleges, and industrial training programs with a professional, yet budget-priced, microscope for the study of opaque objects.

▶ A new automatic burette with only one opening to the atmosphere (instead of two or three as in conventional designs) has just been announced by the Scientific Glass Apparatus Co., Inc., Bloomfield, New Jersey. According to the manufacturer, this construction permits better control of the environment above the liquid in the reservoir, reducing the possibility of contamination.

▶ A new approach to small capacity pumping applications involving liquids or gases has been achieved in the design of the Roll-Flex laboratory pump recently introduced by Cole-Parmer Instrument & Equipment Co., 7330 N. Clark St., Chicago 26, Illinois.

▶ The Model H598 Tritium Monitor utilizing one electrometer tube and all transistorized circuitry thereafter has four linear ranges: 0–100uc/m3; 0–1,000 uc/m3; 0–10,000uc/m3; and 0–100,000 uc/m3. The meter reads directly in tritium concentration; however, other radioactive gases can be monitored qualitatively. For additional information write: Radiation Technology, Inc., 657 Antone St., N. W., Atlanta 18, Georgia.

▶ A new dosimetric system, that measures both fast neutrons and gammas (or x-radiation) in rads, is now available from Atomic Accessories, Inc., 811 W. Merrick Rd., Valley Stream, New York. Model NDS-1000 Neutron Dosimetric System consists of two direct-reading, fountain-pen sized, pocket dosimeters. One is a tissue-equivalent type, for fast-neutron and gamma doses. The second is a neutron-insensitive dosimeter, for gamma doses only.

▶ Laboratory Construction Co., 8811 Prospect St., Kansas City 32, Missouri, announces a new product in its line—a fiber-glass fume hood. Called the Fiberglass "47" Fume Hood, this new laboratory product is molded of resistant polyesters over fiberglass, making possible a fume hood that is light, chemically resistant and attractive, yet comparatively low in price.

▶ Glas-Col Apparatus Co., 711 Hulman St., Terre Haute, Indiana, announces its new Kjeldahl distillate heating mantle for pilot plant and laboratory use. Because wattage requirements for distilled aqueous solutions are high, its six heating mantle units are made from quartz fabric for safe, dependable operation. Heat input for each unit can be individually controlled.

(*Continued on page A618*)

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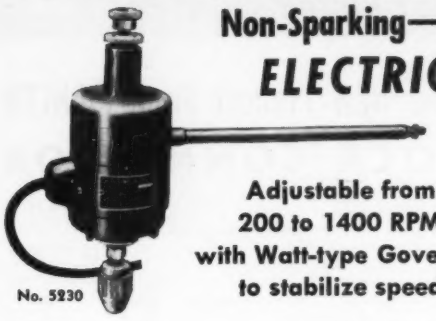

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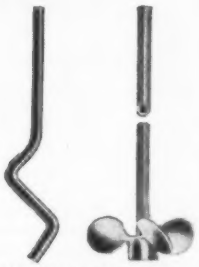

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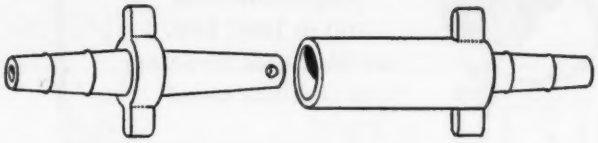

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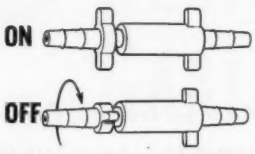

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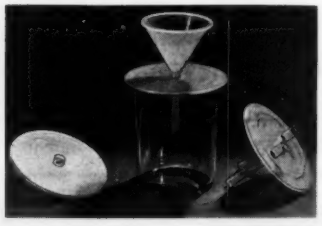

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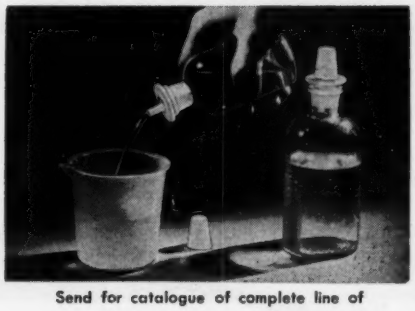

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Out of the EDITOR'S BASKET

▶ Labline, Inc., 3070 W. Grand Ave., Chicago 22, Illinois, has introduced what is claimed to be the only line of baths on the market with magnetic, rotary stirring action. The "Magnestir" baths, as they are called, come in four sizes, from $6^1/_2$ in. L × 12 in. W × 7 in. deep to 36 in. L × 18 in. L × 18 in. W × 9 in. deep. Magnetic stirring (Pats. Pending) is achieved with only one moving part.

▶ A new dry chemical extinguishing agent effective on Class A, B and C fires, has been introduced by Ansul Chemical Co., Marinette, Wisconsin. Known as "Foray," it is the first multipurpose dry chemical to be offered in a complete line of hand portable extinguishers.

▶ A slide-rule-type high vacuum calculator useful in determining pump or chamber size, as well as time and pressure, for high vacuum systems has been developed by Consolidated Vacuum Corp., 1775 Mt. Read Blvd., Rochester 3, New York. The calculator retails for two dollars.

▶ Industrial Instruments, Inc., 89 Commerce Rd., Cedar Grove, Essex County, New Jersey, has just announced the addition of a new Solu Meter to its line of Solu Bridges designed for laboratory and plant solution conductivity measurements. When used with suitable conductivity cells, this compact instrument can be used for the measurement of conductivities ranging from distilled and demineralized water to concentrated acids and bases.

▶ Ohio Chemical has introduced a new model of its pressure-compensated flowmeter especially designed for laboratory use. A variety of calibrations are available for service with different gases including argon, carbon dioxide, helium, oxygen and compressed air. For additional details, write Ohio Chemical & Surgical Equipment Co., 1400 E. Washington Ave., Madison 10, Wisconsin, and request Catalogue Sheet No. 2453A.

▶ Nuclear-Chicago Corp., 359 E. Howard Ave., at Nuclear Dr., Des Plaines, Illinois, is now marketing a Neuteron Generator system which consists of (1) a small, mobile accelerator, (2) a 150,000 volt power supply, and (3) a remote-control console. A radio-frequency type ion source with an atomic/molecular ratio of 90% is used to deliver beam currents in excess of one milliampere. Controllable yields to 4×10^{10} fast neutrons per second may be obtained. A getter-type pumping assembly provides a clean vacuum of 10^{-7} mm. Hg. Applications of the Neutron Generator include activation analysis, process control, student training, and basic radiation research where an economical high flux neutron source is desired.

▶ Development and production of the

(*Continued on page A620*)

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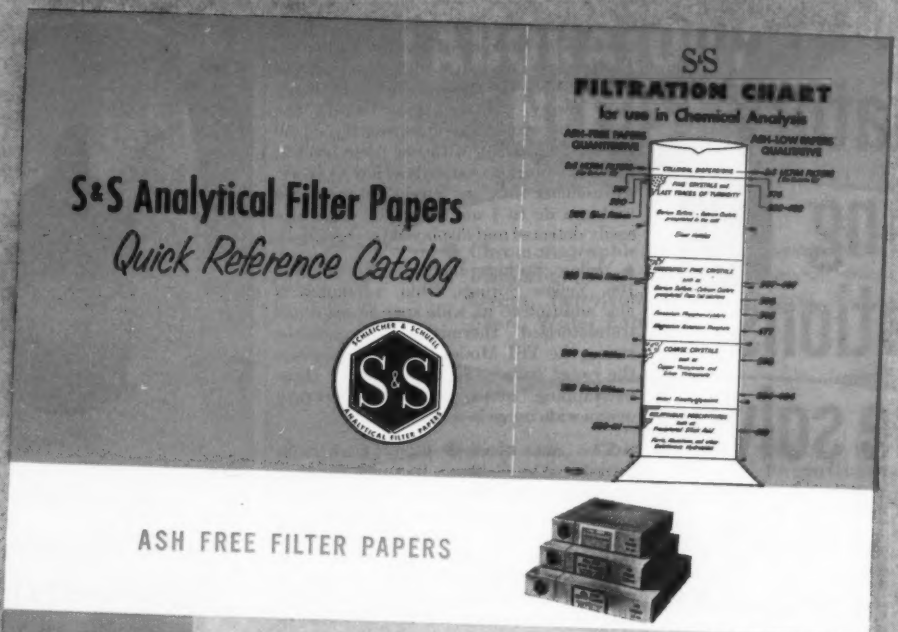

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Out of the EDITOR'S BASKET

most sensitive oscilloscope commercially available is announced by the Industrial Electronics Division, Allen B. DuMont Laboratories, 750 Bloomfield Ave., Clifton, New Jersey. Termed Type 403-B, the new oscilloscope has a sensitivity of 50 microvolts per cm, with low noise level to permit resolution of signals as low as 10 uv. Phenomena that are barely determinable within a dc to 1 mc frequency range are easily detected and displayed by the 403-B.

▶ Yellow Springs Instrument Co., Box 106, Yellow Springs, Ohio, announces a new addition to its wide span multi-range transistorized thermistor thermometer series, the YSI Model 42SL which covers the range from −80 to +40°C in three overlapping steps. Absolute accuracy over a wide range is ±0.5°C.

▶ Two new spectral filters have been introduced by Coleman Instruments, Inc., 42 Madison St., Maywood, Illinois, for use with the Coleman Flame Photometer in laboratory determinations of magnesium and lithium. These filters extend the range of the instrument far beyond its most common applications in medical and clinical laboratories for the analysis of sodium, potassium and calcium in body fluids.

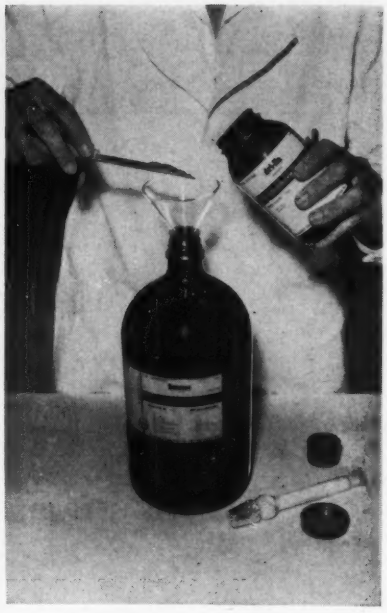
▶ A half million volt electrostatic generator is combined with a high energy particle accelerator in the new low-cost Rayotron now being marketed by Atomic Laboratories, 3086 Claremont Ave., Berkeley 5, California. As a particle accelerator, the Rayotron generates high-energy X-ray experiments. The Compton Effect and Einstein's Photoelectric Effect are a few of the physical phenomena that can be dramatically produced. The Rayotron's X-rays also register clearly in cloud chambers and Geiger counters.

▶ Interference filters for precision spectral analysis now are being marketed in boxed sets, individually calibrated by Optics Technology, Inc., 248 Harbor Blvd., Belmont, California. The Spectrocoat filters are selected to isolate visible spectrum wavelengths important in laboratory and industrial work. Each set contains ten interference filters covering the range from 404 to 706 millimicrons (K to Ba lines), four neutral density filters and one continuously - variable wedge filter covering the 400–750 millimicron range. All are of rugged construction, impervious to fume oil.

▶ A new ingenious method for dispensing, titrating and decanting liquids has just been introduced by Labline, Inc., 3070 W. Grand Ave., Chicago 22, Illinois. The new unit, called the Rapid-Saf Pipetter, eliminates the need—and the danger—of using the mouth to draw liquids into pipettes. Operation is simple, fast and accurate—permits one hand operation with finger tip micrometer control.

▶ A newly available Microtitrator designed by the Manostat Corp., 26 N.

(*Continued an page A622*)



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New Literature

● The complete line of scientific instruments, apparatus and equipment, manufactured by Labline, Inc., 3070 W. Grand Ave., Chicago 22, Illinois, and its subsidiaries, has been incorporated into a new illustrated catalogue available to all scientific and purchasing department personnel.

● The Audio-Visual Center, Division of University Extension, Indiana University, Bloomington, Indiana offers *Films for Junior and Senior High School Biological and Physical Sciences.* This is a descriptive index of 16 mm motion pictures arranged according to subject matter and available from their office.

● Application *Bulletin 90-26-07*, published by Fischer & Porter Co., 915 Jacksonville Rd., Warminster, Pa., outlines the construction of a "build-it-yourself" pH electrode chamber designed to minimize the maintenance problems common to many conventional immersion- and flow-type electrode housings.

● Just off the press is the new edition of *Lablog*, supplement to Will's General Catalog 7 available from Will Corp., Box 1050, Rochester 3, New York. Highlighting the new issue is complete information on the Will Biosonik Cell Fracturing Apparatus, which utilizes pretuned ultrasonics at 20 kc to effectively disrupt cellular material without inactivating the enzymatic protein.

● Kimble Glass Co., Toledo 1, Ohio, offers a four page folder describing a do-it-yourself machine for hot cutting glass tubing, including an engineering drawing.

● *Getting Acquainted with the Microscope* by Dr. Julian D. Corrington provides both student and teacher with a thorough introduction to the basic instrument for life science courses. It is available for fifty cents a copy (science department heads may obtain a free copy), from Bausch and Lomb, Inc., Rochester 2, New York.

● A technical brochure on the newly-announced A-60 Analytical NMR Spectrometer System has been published by the Instrument Division, Varian Associates, 611 Hansen Way, Palo Alto, California. The 16-page brochure, illustrated with photographs and technical diagrams, includes discussions of range of applications, ease of operation and maintenance, theory of A-60 operation with special emphasis on the unique Varian Field Frequency Control System, and performance specifications.

● A brochure describing the Model 202 Spectrophotometer, a new low-cost instrument operating in the ultraviolet and visible region of the electromagnetic spectrum, has been published by the Instrument Division, Perkin-Elmer Corporation, Norwalk, Connecticut.

● A new descriptive bulletin on the Beckman DB Ultraviolet Spectrophotometer is available from the Scientific and Process Instruments Division of Beckman Instruments, Inc., 2500 Fullerton Rd., Fullerton, California. The 16-page brochure describes major features, outlines specifications, and explains the optical system and operating principles of the double-beam instrument. Numerous curves illustrating the DB's capabilities also are included.

● An illustrated sixty-four (64) page, two-color miniature *Catalogue No. 600*, covering Blue M Electric Company's complete line of electric ovens, furnaces, magni-whirl baths, environmental cabinets and related temperature controlled equipment for the laboratory, is now available from the company at 138th and Chatham Sts., Blue Island, Illinois.

● The following is available *by number* from the U. S. Government Printing Office, Division of Public Documents, Washington 25, D. C. Catalogue No. I 19.3:1084-H, "An Evaluation of Whole Order, one half Order and one third Order Reporting in Semi-qualitative Analysis." 15 cents.

(*Continued on page A625*)

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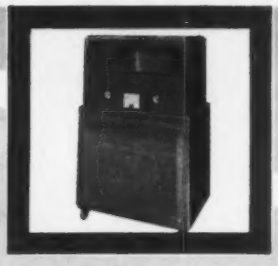

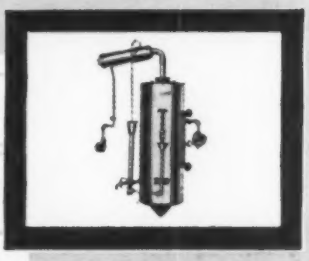

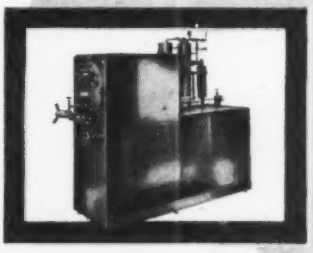

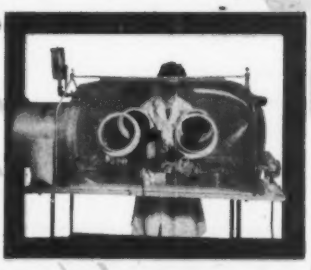

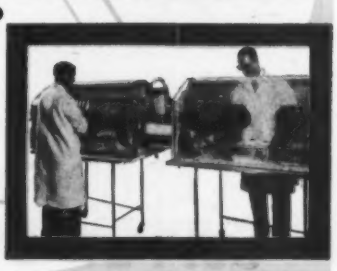

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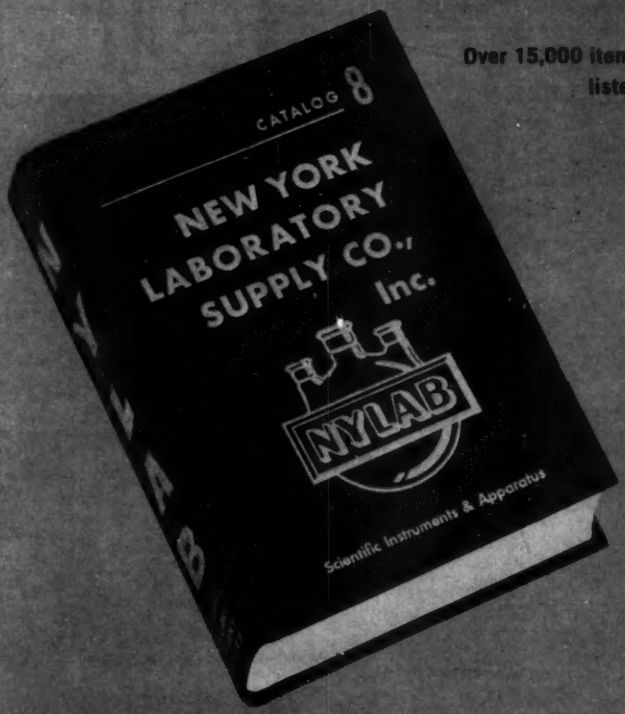

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● The Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C., offers:

Atomic Energy Levels in Crystals, by John L. Prather, National Bureau of Standards Monograph 502, issued February 24, 1961, 84 pages.

Measurement of Absorbed Dose of Neutrons and of Neutrons and Gamma Rays (Recommendations of the National Committee on Radiation Protection and Measurements) National Bureau of Standards Handbook 75, issued February 3, 1961, 86 pages, 35 cents.

Corrected Optical Pyrometer Readings, by D. E. Poland, J. W. Green, and J. L. Margrave, National Bureau of Standards Monograph 30, issued April 21, 1961, 74 pages, 55 cents.

● Radiation Equipment and Accessories Corp., 665 Merrick Rd., Lynbrook, New York, offers a new loose leaf type catalogue listing scalers, educational equipment, transfer systems, preparation equipment and miscellaneous crystals for special jobs.

New Chemicals

◇ Several polyesters useful for analysis of steroids are now available from Applied Science Laboratories, Inc., State College, Pennsylvania. Included are neopentyl sebacate and neopentyl adipate. These materials have been purified and are noted for their thermal stability at temperatures to 250°C.

◇ Hydripills are new light-weight convenient source of hydrogen for industrial and laboratory applications that have been developed by Metal Hydrides Inc., 12-24 Congress Street, Beverly, Massachusetts. A tableted mixture of sodium borohydride and cobalt chloride, Hydripills will react catalytically with water to produce hydrogen. Rates of hydrolysis are rapid.

Miscellany

★ The combined Regional Meeting of The Southwest and Southeast Sections of the ACS will be held in New Orleans December 7, 8, and 9, 1961. The technical program chairman, Dr. Jack Carlton of Louisiana State University, New Orleans has announced the following program:

Chemical Eduation Symposium: "Curricula Trends in Chemical Eduation"—Winston R. de Monsabert, Loyola University.

Organic Chemistry Symposium: "New Synthetic Methods"—Albert F. Meyers, L.S.U.

Inorganic Chemistry Symposium: "Inorganic Nuclear Chemistry"—Hans B. Jonassen, Tulane University.

Joint Symposia Between Physical Chemistry and Spectroscopy and Gas Chromatography: "Molecular Spectroscopy"—Thomas Fagley, Tulane University (Physical Chemistry), and Robert T. O'Connor, Southern Utilization Re-

(*Continued on page A626*)

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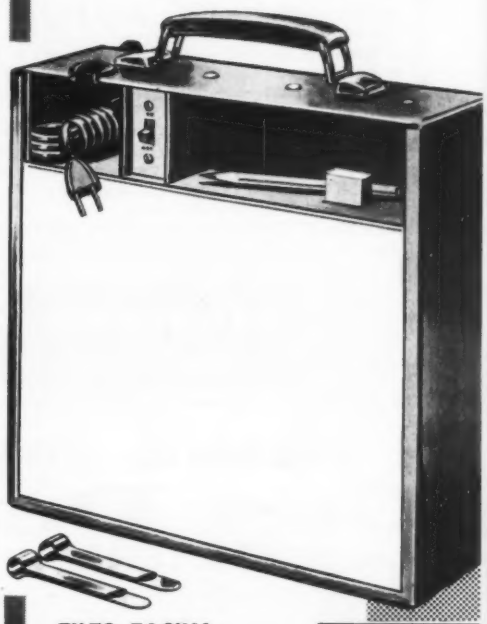

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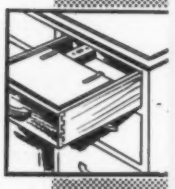

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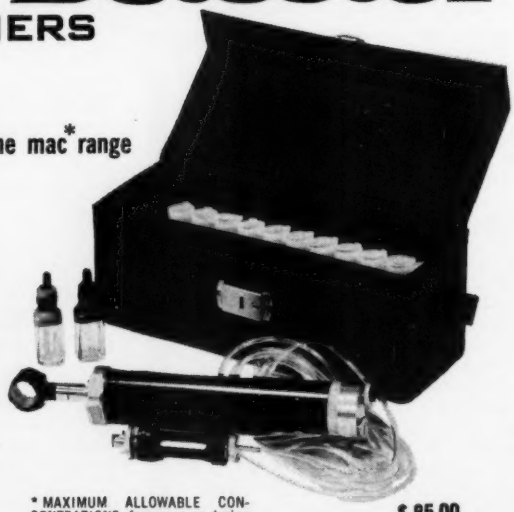

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search and Development Division (Spectroscopy and Gas Chromatography).

Biochemistry Symposium: "Biochemical Studies in Mental Diseases"—Donald G. Davis, Jr., L.S.U.

Joint Symposium Between Inorganic Chemistry and Industrial and Engineering Chemistry: "Catalysis"—Hans B. Jonassen, Tulane University (Inorganic Chemistry), and Calvin C. Rolland, American Cyanamid (Fortier Plant), Avondlae, Louisana (Industrial and Engineering Chemistry).

★ The Advertising and Sales Promotion Dept., Corning Glass Works, Corning, New York, offers two new movies "Pyrex brand 'double - tough - Drainline'" and "How to Cut and Bead Pyrex Brand Pipe."

★ "Hard to Handle" is the title of a new 16 mm color, sound movie on the development of equipment to meter and pump hard to handle liquids. For information write the Process Equipment Div. of Lapp Insulator Co., 125 Hall St., LeRoy, New York.

★ A recent redetermination of the half life of carbon 14 by the National Bureau of Standards yielded a value of 5760 years. The value previously accepted was 5568 years, a weighted average of three experimentally determined values. This discrepancy will affect the dating of such artifacts as the Dead Sea Scrolls.

★ Final plans have been announced for the 11th Annual Instrument Symposium and Research Equipment Exhibit to be held October 9–12 and October 10–13, respectively, at the National Institutes of Health in Bethesda, Maryland, a suburb of Washington, D. C. The NIH is the principal research center of the U. S. Public Health Service.

★ The 1961 Eastern Analytical Symposium will be held at the Statler Hilton Hotel in New York City on November 15, 16, and 17, 1961. It is sponsored by the Analytical Groups (ACS) of the New York and North Jersey Sections, four sections of the Society for Applied Spectroscopy (New York, Delaware Valley, New England, and Baltimore-Washington), and the Metropolitan Microchemical Society.

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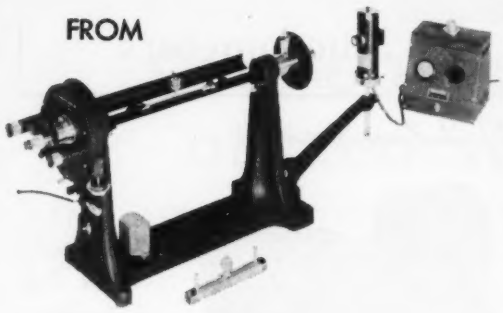

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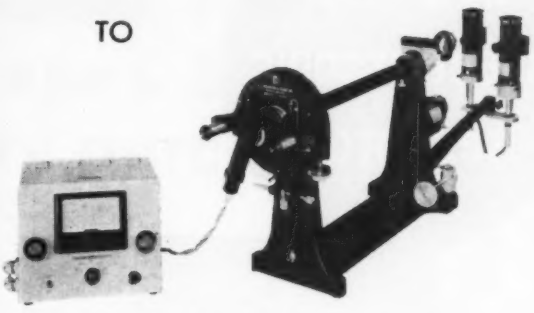

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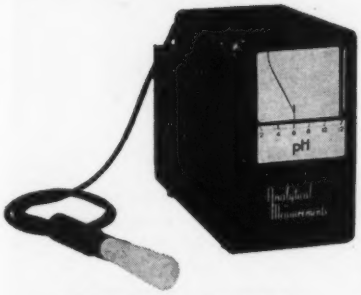

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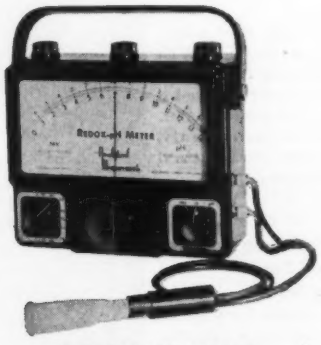

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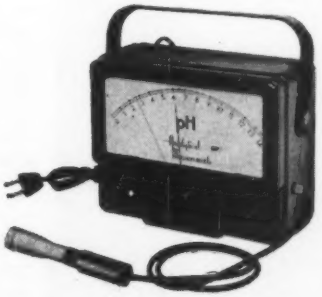

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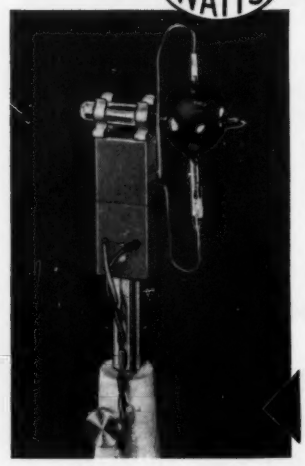

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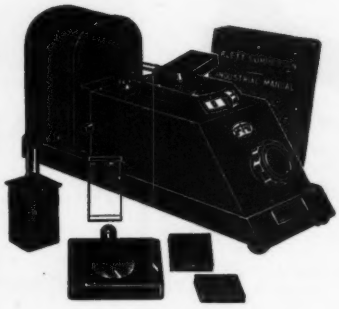

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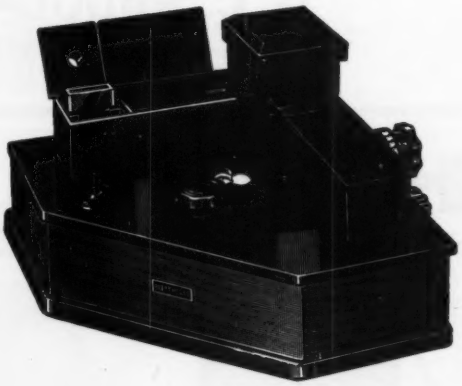

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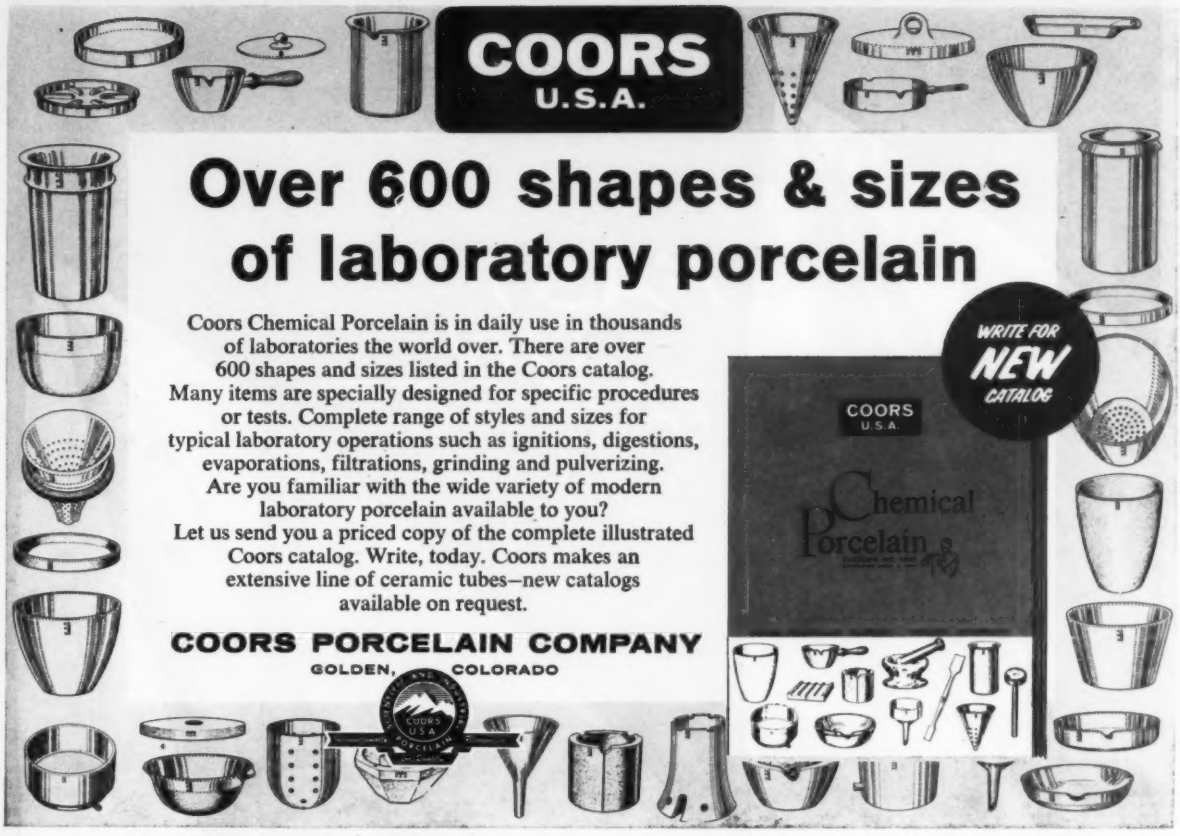
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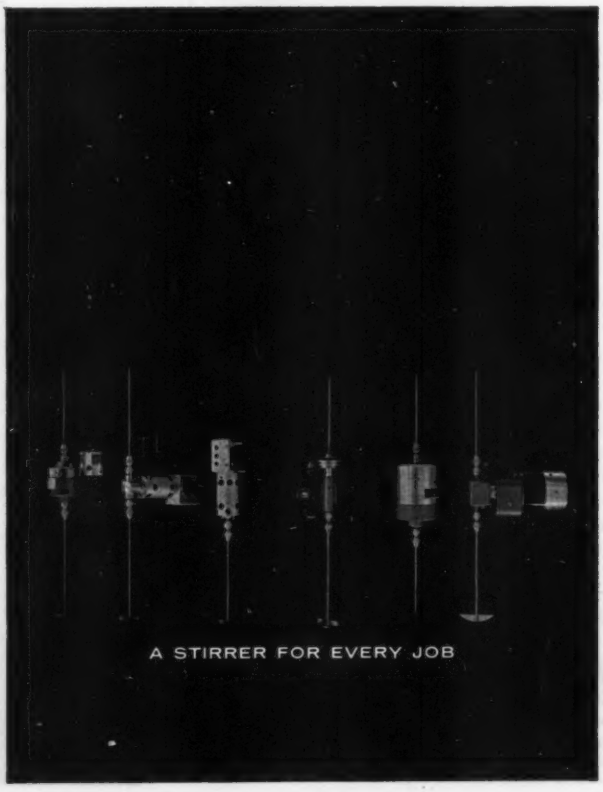

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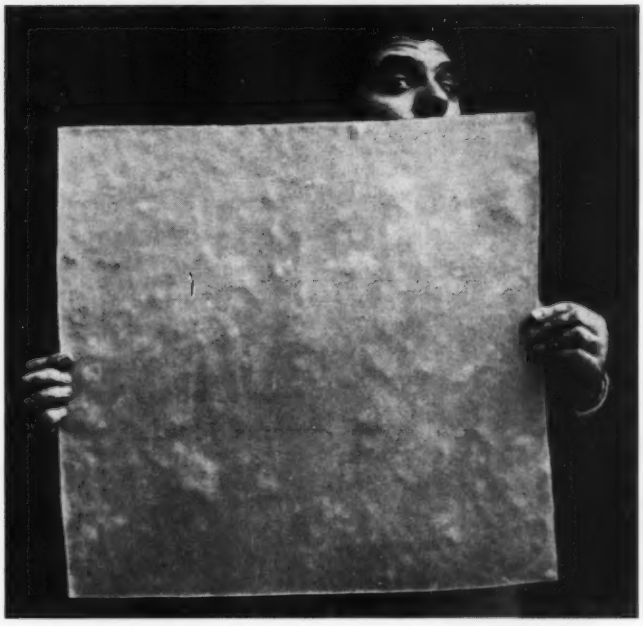

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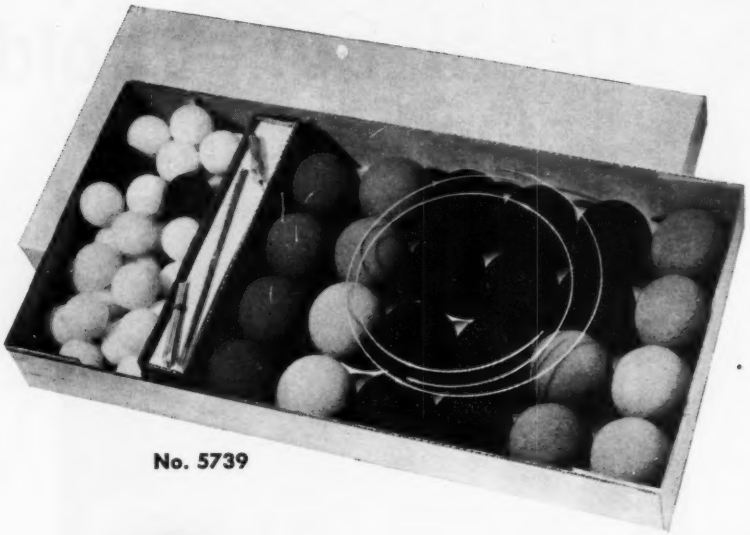

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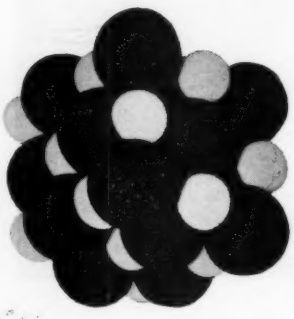

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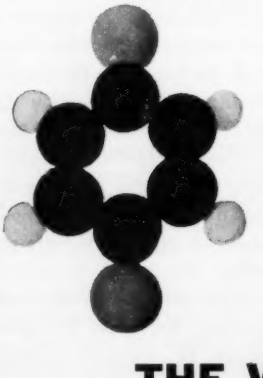

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EDITORIALLY SPEAKING

Santa Claus may seem to be an odd topic for remarks in September, but here he is. Let's see what happens to our ideas about the old boy in the white beard and red suit during the two decades of our lives from four to twenty-four; then let's see what happens to some of the ideas our students get about chemical concepts during about twenty weeks of their early lives as chemists.

To the toddler, Santa is very real. He does not even need definition; he *is*. It does not occur to the small child to wonder about the relationship between Santa's girth and the chimney dimensions. Not even the multiplicity of impersonations clouds the simple image. Then sophistication sets in. The first grader knows, and he can be bombastic in proving, that "there ain't no Santy Claus." Adolescence calms him down on the subject, but all his very logical use of information increases his disdain of the earlier fantasy. Then finally as an adult he becomes a parent. When a new generation of toddlers appears, Santa reemerges as a mighty, profound idea. The famous letter to Virginia is not juvenile literature. It speaks to the mature, experienced person who reads more than a definition in so many words. Santa Claus is an idea, not a person.

So it is, by analogy, with many of the ideas chemistry teachers will be trying to plant in young minds, starting in September. How many "Santa Claus concepts" there are in introductory chemistry! Acids and bases, oxidation and reduction, electronic structure, titration and equivalence, and equivalent weight are examples. Let us examine a few.

Earliest definitions should make sense to the inexperienced. The first acid a neophyte chemist knows about is something which turns litmus red and neutralizes an hydroxide. So with the base at the "Santa-is-a-jolly-man" level. It does something: it neutralizes an acid. But if "base" means "OH somewhere in the formula," disillusionment is sure to set in when he finds that CaO, Na_2CO_3, and even acetate ion will neutralize acids. Then he is in the crucial "There-ain't-no-Santa Claus" stage. He needs to have some broader view to make sense out of a lot of additional information. It is not enough merely to shatter his earlier image; a replacement is needed. The Brönsted-Lowry conceptual scheme for acid-base phenomena correlates even the subtler observations such as amphoterism of some hydrated cations with his increasingly more sophisticated appreciation of structural features. Finally, the mature adult view of "Santa" is the Lewis picture which makes even the Brönsted acids and bases appear to be simplified special cases of a far more inclusive pattern of phenomena.

The Santa parallel also fits the process of developing a full appreciation of redox. "Combination with oxygen" for oxidation can last only a short time. Soon "increase in positive oxidation number" is needed as a replacement. "Loss of electrons" comes along next. Finally an appreciation of the distinction between on-the-paper balancing of an equation and the in-the-beaker mechanism of the reaction signifies having attained adulthood.

The analogy points up some important warnings for the teacher. Allowing a child to hold on to juvenile views may be comfortable but is ludicrous. Almost equally silly is an attempt to force a child's mind to appreciate an adult idea. Balanced between is a gradual growth in presentation from the simple to the complex, from the isolated to the all-inclusive. So it is with the beginning chemistry student and the major concepts of chemistry. Furthermore, once the growth has started and is sympathetically nurtured, it can be gratifyingly rapid.

Santa Claus in September? All year long, too!

John C. Bailar, Jr.
University of Illinois
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Chemical Education—Then and Now

My original intention, in planning this talk, was to describe the changes which have taken place during the last 80 or 90 years in all branches of chemical education, but I quickly found that these changes were far too numerous to cover in a single lecture. I therefore decided to limit my study to developments in general college chemistry, since I am most familiar with this branch of the subject. Regrettably, I must omit the teaching of organic chemistry, in which tremendous strides have been made in the last few years, and physical chemistry, which has lived almost its entire life in the period under review. I regret most the necessity to omit discussion of the recent exciting developments in high school chemistry, in which perhaps the greatest advancements of all are being made. A few years ago, the typical high school book was simply an abridgment of a corresponding college text, but now, secondary school courses and texts are leading the way in new methods of teaching chemistry. The tremendous development of chemical education in the high schools has been sparked in large measure by the NSF through its summer institutes and its sponsorship of the Chemical Bond Approach and the Chemical Education Materials Study to the teaching of chemistry in the secondary schools. The high school course on film, which was prepared under the guidance of a committee appointed by the ACS, and the Continental Classroom Modern Chemistry course, which has reached half a million TV viewers, have also played important roles.

As part of my preparation for this talk, I have read (or in some cases, reread) several college textbooks of general chemistry. Among these were the texts written by Roscoe[1] in 1877, by Remsen[2] in 1906, by Holleman[3] in 1916, the Smith-Kendall[4] book published in 1926, and several of the more recent texts. While these do not cover the whole field, they are probably representative of the textbooks of their times. At least, they show the trends of thought.[5]

The 1961 Scientific Apparatus Maker's Award in Chemical Education address given before the dinner meeting of the Division of Chemical Education at the 139th Meeting of the ACS, St. Louis, March 28, 1961.

[1] ROSCOE, HENRY E., "Lessons in Elementary Chemistry," new ed., Macmillan and Co., London, 1877.

[2] REMSEN, IRA, "An Introduction to the Study of Chemistry," 7th ed., Henry Holt and Co., New York, 1906.

[3] HOLLEMAN, A. F., "A Textbook of Inorganic Chemistry," issued in English in cooperation with H. C. Cooper, 5th English ed., John Wiley and Sons, New York, 1916.

[4] KENDALL, JAMES, "Smith's College Chemistry," The Century Company, New York, 1926.

[5] I wish to express my thanks to Professor Virginia Bartow and Miss Ruth Power for their help in locating suitable articles and books.

Cannizzaro (1861): "Teach Theory"

To see how much chemical education has changed in the past hundred years, one has only to read the Faraday Lecture which Cannizzaro delivered before the Chemical Society in 1861,[6] in which he extols the use of the atomic theory in teaching chemistry. He says, in part

> Is it desirable, in explaining all that relates to the meaning, use, and value of chemical symbols, formulas, and equations, to dispense with the atomic and molecular theory as a superfluous and dangerous speculation, and confine ourselves wholly to the empirical laws on which the formulas are based?
>
> After many trials in the course of my teaching, I have come to the conclusion, that not only is it impossible to eliminate the atomic theory altogether, but, moreover, that in order to arrive at this theory, it is not desirable to follow the long and fatiguing road of induction. On the contrary, it is better to get to it as quickly as possible, by one of those short cuts which the human mind often takes in order to raise itself quickly to a height from which the relations between phenomena can be discerned at a glance. For, whether it be due to the intrinsic nature of chemical phenomena and their relations to our ideas, or to the imperfection of the science in its actual state, certain it is that we have not yet succeeded in enunciating the empirical laws of chemical action with that clearness and precision which has been attained in other cases, as, for example, in the enunciation of Kepler's laws relating to the movements of the planets. Hence it follows that the so-called laws of definite, reciprocal, and multiple proportions, of simple and compound equivalents, and of gaseous volumes, when unaided by the atomic and molecular theory, do not penetrate sufficiently into the minds of the majority of students, and do not become fixed there, but remain like floating isolated objects, without any connection between them.
>
> On the other hand, to embrace all these laws in a single glance, and with clear and distinct vision, it is sufficient to regard the phenomena as if each element were in reality made up of homogeneous atoms, having a special and constant weight, and as if the juxtaposition of these atoms gave rise to the molecules, both of simple and of compound bodies, equal numbers of these molecules being always contained in equal volumes of perfect gases, under given conditions of pressure and temperature—that is to say, it is sufficient to comprehend the atomic and molecular theory in the state of development which it has actually attained.
>
> To the different types of mind which are met within a school of chemistry, this theory affords the clearest, shortest, most exact, and most accessible summary of all that relates to the origin, meaning, value, and use of empirical formulas and of equations. It ought, therefore, to be introduced into the teaching of chemistry at an early stage, even if we are disposed to regard it merely as a convenient fiction or artifice to aid the mind in seizing the relations between facts.

Laboratory versus Demonstration

One of the topics which interested me most during this study was the recurring debate over the relative merits of the lecture demonstration method of teaching chemistry and the use of individual laboratory

[6] CANNIZZARO, STANISLAO, *Chem. News* **3**, 245 (1861).

work. In the early days of chemical instruction in this country students did no laboratory work at all. The elder Silliman is quoted as having remarked

> Many times I have said to those who as novices have offered aid to me that they might come and see what we were doing, and I much prefer that they should do nothing; for then they would not hinder me and my trained assistants, nor derange nor break the apparatus.[7]

Yet, it was Silliman Junior who, in 1842, established the first college laboratory course in chemistry in the United States at Yale University. He and his colleagues must have felt very strongly on the subject, for the professors not only paid out of their own pockets for the upkeep of the laboratory and for the chemicals which the students used, but had to pay their own salaries as well. With this precedent, it is not surprising that chemical education via the laboratory method grew rather slowly. Even thirty years later, when Remsen asked for funds to equip a chemical laboratory at Williams College, he was told that Williams was not a manual training school. However, by 1900, laboratory work was almost universal, and its superiority over the lecture demonstration method seemed to have been established. A few years later, however, the efficiency of laboratory instruction again came into question and in the early 1930's, several studies were made of the relative merits of the two systems. The authors of some of these studies were quite critical of the use of individual laboratory experiments in chemical instruction[8] and several universities established courses in which only the lecture demonstration method was used. Most of these courses were discontinued after a few years. Now, however, the lecture demonstration method is again being advocated and several colleges and universities are once more introducing demonstration courses. While instruction through individual laboratory work certainly has many faults, its superiority is apparently conceded by most teachers of general chemistry. It is significant that the growth of demonstration courses 30 years ago came at a period when colleges and universities were suffering severely from lack of money, and the present interest in demonstration courses comes at a time when college laboratories are so crowded that students are being turned away. Under normal conditions, the method of individual experimentation seems to prevail.

Consistencies and Changes in Content

There are a few topics in general chemistry which have changed very little during most of the past century. For example, until recently almost all textbook authors included long discussions on combustion and on the structure of flame. Perhaps these were inspired by Faraday's lectures on "The Chemical History of a Candle," which were published a century ago, but their prominence in chemical education is doubtless due, as well, to the fact that combustion is one of the most familiar of all chemical processes. Faraday's lectures covered far more than the chemistry involved in the burning of a candle. They included such topics as the brightness of flame, the necessity of air for combustion, the fact that water is produced, the nature of water, hydrogen, the presence of oxygen in the air, the properties of the atmosphere, the formation of carbon dioxide by combustion, carbonic acid, carbon, charcoal and coal gas, and finally, respiration. These topics, along with the chemistry of the halogens and the metals, corrosion, valence, and the nature of acids and bases, have held prominent places in all courses in general chemistry during most of the past century; but in recent books, all except the last two have occupied much smaller roles.

Over the entire period which I am reviewing, there has been a constant effort on the part of teachers and textbook writers to teach the principles of chemistry, rather than to allow the students to rely upon memory. Efforts to explain valence and the structure of molecules were apparent from the very beginning. For example, Professor George F. Barker wrote in the preface to his "Textbook of Elementary Chemistry,"[9] published in 1870

> Within the past ten years chemical science has undergone a remarkable revolution. The changes which have so entirely altered the aspect of the science, however, are not, as some seem to suppose, changes merely in the names and formulas of chemical compounds; for in this, the science is but returning to principles long ago established by Berzelius. They are changes which have had their origin in the discovery, first, that each element has a fixed and definite combining power or equivalence; and second, that in a chemical compound the arrangement of the atoms is of quite as much importance as their kind or number. The division of the elements into groups, according to the law of equivalence, necessitated a revision, and in some cases an alteration, of their atomic weights; while, in obedience to the second law, molecular formulas were reconstructed so as to express this atomic arrangement. The importance of these laws cannot be over-estimated. By the former, all the compounds formed by any element may be with certainty predicted; by the latter, all the modes of atomic grouping may be foreseen, and the possible isomers of any substance be predetermined. Instead, therefore, of being a heterogeneous collection of facts, chemistry has now become a true science, based upon a sound philosophy.
>
> The first part of this book is intended to be an elementary treatise upon Theoretical Chemistry. It aims to present the principles of the science as they are held by the best chemists of the day, upon a new plan of treatment which the author has found simple and satisfactory in his own teaching.
>
> The second part of the book contains the facts of Inorganic Chemistry, arranged systematically under appropriate heads. To as great an extent as seemed desirable, theory has been applied to explain the formation and properties of compounds. The unsatisfactory classification of the elements into metals and metalloids is discarded, and they are arranged electro-chemically, from negative to positive.

In reviewing Professor Barker's book, Professor Charles F. Chandler[10] quoted at length from this preface and added

> The aims of the author are fully realized. Professor Barker has succeeded admirably in condensing in the space of 67 small pages the modern theories of chemistry in logical sequence and in the clearest language.

It is obvious that theoretical chemistry was less extensive in 1870 than in 1961.

In recent years, chemistry has lost some of the aspect of general education which it held in earlier days. In the older textbooks, many pages were devoted to the philosophy of science, the history of chemistry,

[7] Quoted by Browne, C. A., J. Chem. Educ., 9, 696 (1932).
[8] Payne, V. F., J. Chem. Educ., 9, 932, 1097, 1277 (1932).

[9] Barker, George F., "A Textbook of Elementary Chemistry, Theoretical and Inorganic," C. C. Chatfield and Co., 1870.
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the derivations of the names of substances, and the influence of chemistry on our national economy and our international relationships. Many teachers of the "modern" school—especially the younger ones—seem to feel that these topics are not an integral part of a course in chemistry. When I recently mentioned to some of my younger colleagues that in my lecture I had taken time to point out that because of the Haber process, we are able to produce so much food that we can't use it all and that we literally give away millions of tons of it, they were shocked, and I could not convince them that this was worth class time. The older texts, of course, had much more room for discussion of the history of chemistry and the development of ideas than do the modern books, for there was much less chemistry to be learned then than now. For example, many substances which are now well known had not yet been discovered. Roscoe's text lists 63 elements; now we recognize 103. Hydrazine, the fluorides of sulfur, phosphorous-nitrogen compounds, and thousands of other familiar materials were quite unknown to the textbook writers of a few decades ago. Again, many chemical processes have been developed in recent years. The arc process for the manufacture of nitric acid has come and gone. The Ostwald and Haber processes were developed only in this century. The flotation process for the concentration of ores and electrolytic methods of making chlorine on an industrial scale are of comparatively recent origin. Some of these topics, which seemed so exciting a generation ago, have been almost squeezed out of general chemistry by new theoretical developments, and receive only a few lines in the recent textbooks. In one 1959 book, the Frasch process for mining sulfur and the Dow process of obtaining bromine from the sea each receive six lines.

Recognition of Mechanism and Structure

It is surprising to find that textbook authors are frequently inconsistent and present quite different ideas about the same subject within a single book. For example, although he was well aware of the composition and properties of ozone, Roscoe included the equation $2KClO_3 = 2KCl + O_3$ in his text. On another page of the same book, he wrote $2KClO_3 = KClO_4 + KCl + O_2$. (The latter equation appears also in Remsen's book, which was written some thirty years later.) It is, of course, quite incorrect, for it implies that in the decomposition of $KClO_3$, half of the material is converted to $KClO_4$ and the other half to KCl. Holleman wrote two different mechanisms for the reaction of copper with concentrated sulfuric acid:

$$Cu + H_2SO_4 \rightarrow CuSO_4 + H_2$$

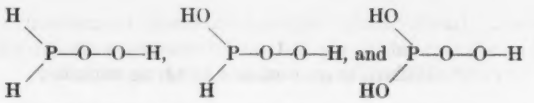
$$H_2 + H_2SO_4 \rightarrow SO_2 + 2H_2O$$

and

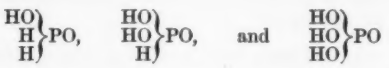
$$Cu + H_2SO_4 \rightarrow CuO + SO_2 + H_2O$$

$$CuO + H_2SO_4 \rightarrow CuSO_4 + H_2O$$

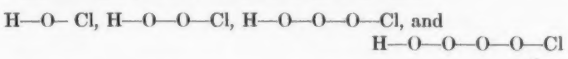
In an attempt to maintain a constant valence of three for phosphorous and to show the varying number of replaceable hydrogen atoms in its oxy acids, Roscoe wrote the formulas for hypophosphorous, phosphorous, and phosphoric acids as



In the same book, however, he wrote the formulas for these substances



which is much as we would write them now. It should be said to Roscoe's credit that, in line formulas, he wrote the formula for hypophosphorous acid as HPH_2O_2, indicating that only one hydrogen atom is replaceable by metals. It is unfortunate that most modern authors have not adhered to this very logical plan. Roscoe's formulas for the oxyacids of chlorine were



He pointed out that the stability of acids increases with increasing members of oxygen atoms, but he did not explain why $HClO_5$ and $HClO_6$ do not exist as even more stable entities than $HClO_4$.

Until recent years, our knowledge of reaction mechanisms developed slowly and found its way into some of the textbooks of general chemistry even more slowly. For example, the theory that the electrolysis of brine liberates metallic sodium (which then reacts with the water to give sodium hydroxide and hydrogen) was advocated in some textbooks fully 20 years after other authors had abandoned it. As late as 1930, the idea of "nascent" elements was still being widely taught, although there was much evidence to refute it. Kendall, in his 1926 revision of Smith's College Chemistry, wrote several paragraphs pointing out the fundamental errors in the theory of the nascent state.

Although much experimental evidence had been accumulated on molecular structure and crystal structure as long as 45 years ago, these subjects played a very minor role in most textbooks of general chemistry until the early 1940's. The most recent texts quite properly spend a great deal of time on these subjects, and we find in them detailed discussions of bond lengths, bond angles, and the stereochemical arrangements of atoms in molecules and ions. In fact, atomic and molecular structures have become almost the keystone of general chemistry, and nearly all of the recent books emphasize over and over that the properties of compounds hinge upon the atomic and ionic radii of the elements and their electronegativities.

The theory that there are different kinds of valence bonds was introduced by Werner in 1893 and put into electronic terms by Lewis and Kossel 20 years later. However, this concept didn't reach the textbooks of general chemistry until the middle 1940's. Since that time progress has been rapid, and our ideas of molecular structure are now refined enough that most of us tell our freshmen about resonance, electron density, and π-bonding. The octet theory is still stressed too heavily in most textbooks, but many recent authors discuss the structures of such substances as BF_3, ICl_3, $[Co(NH_3)_6]^{+3}$, NO, and other odd molecules, and the concept of electron deficient molecules as illustrated by B_2H_6. We need increasingly to tell our students that our knowledge is still imperfect, and that 20 years

from now, many of the theories of today will have been found to be quite wrong. Unfortunate as it may seem, we have no way to predict which will stand the test of time and which will fall by the wayside.

The concept of ionization has had a long hard row to hoe, and some teachers did not fully understand it until it was more than 45 years old. One textbook author of the mid-twenties explained in detail that the passage of electric current through a solution does not cause the formation of ions, but that they are formed when the salt dissolves. Kendall, writing in 1926, described the structure of sodium chloride as it had been determined by X-ray analysis, but indicated on another page of the same book that the ionization of salt takes place only when it is dissolved in water. Clearly, in such an atmosphere as this, chemical educators were not ready to teach the theory of the complete ionization of strong electrolytes or the importance of interionic attractions.

Periodic Tables

The history of the periodic table is too well known to justify much discussion here, but it merits passing mention. In common with other chemical educators of his time, Remsen taught that the rare earths, like the rest of the elements, occupied consecutive positions in the main body of the table, which in those days was pictured as having eight vertical columns. This led to the prediction of a triad of platinum metals lying below ruthenium, rhodium, and palladium, but above osmium, iridium, and platinum; the existence of such a hypothetical group is indicated in the table in Remsen's text. Although this belief was soon abandoned, the position of the rare earths in the periodic system remained enigmatic for many years, and until the mid-twenties almost all textbook writers discussed this "defect" of the system. The lack of a suitable place for hydrogen and the reversal of the atomic weights of cobalt and nickel and of tellurium and iodine were also causes for concern. All of these anomalies have now been explained (at least, to our current satisfaction) and the textbook writers are well agreed on the nature of the periodic table except for a few details, such as the definitions of the terms "transition metal" and "lanthanide." Some authors end the first transition series with nickel, some with copper, and some with zinc. It must be very confusing to the student progressing from one course to another and from one textbook to another to find that the teachers themselves cannot agree on the definitions of these terms.

The tremendous amount of information which can be both gained from the periodic table and correlated with it is well illustrated in Professor Sanderson's recent book in which the table is repeated many times, each time illustrating a different property, but each time showing the periodic dependence of properties on atomic structure.

The Future: Emphasize Principles

What of the future? How much stress shall we lay on the philosophy of science, how much on the history of chemistry, how much on chemical principles, and how much on descriptive chemistry? Most college courses in general chemistry are still based largely on inorganic chemistry, but description is rapidly giving way to principles, and a place for descriptive inorganic chemistry must be found at the higher levels of college training. It seems to me that under the present conditions, general chemistry must minimize descriptive chemistry and emphasize principles and the dependence of properties and reaction mechanisms on atomic structure. Students in high school, as well as in college, are learning more inorganic chemistry now than ever before, because they are basing their knowledge on the fundamentals—atomic structure and the periodic table. Yet, we must not fail to emphasize that the principles are established entirely by experimental work, and we must trace the historical development of chemical ideas in order to point the way to future progress. Only when the student builds on an experimental and philosophical background will he have real understanding; only on such a basis can he become a true scientist.

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Electron Paramagnetic Resonance of Free Radicals

Among the many techniques of investigation that have been developed during the last decade, electron paramagnetic resonance (EPR) is one of the most significant for chemists. It is expected to play a role comparable to that played by optical spectroscopy during the first half of this century. It has already proved to be particularly fruitful in two fields: the study of the electronic configuration of the transition metal ions in relation to the crystalline field, and the study of the products of irradiation of matter, especially free radicals and color centers. For example, EPR studies made on iron(II) and iron(III) complexes suggested hypotheses for the mechanism of transport of oxygen by haemoglobin; also, studies on irradiated plastics have led to the explanation that their improved mechanical properties are due to cross-linking between their molecular chains.

This article will not attempt in the limited space available to duplicate the details of the pertinent theory (*1*, *2*, *5*, *7*). Rather it will attempt to suggest the importance of the phenomenon as a means of understanding some of the chemistry of free radicals, particularly their stabilization and recombinations.

Electron Paramagnetic Resonance is based on the fact that atoms, ions, molecules, or molecular fragments which have an odd number of electrons exhibit characteristic magnetic properties. Of the $2n + 1$ electrons in such units, the magnetic moments of n electrons are compensated by the other n paired electrons; the remaining unpaired electron has its magnetic moment uncompensated and the molecule itself has therefore a magnetic moment other than zero. For the sake of simplicity, we shall assume that the magnetic moment of the electron is mainly (if not only) due to its spin. This is not too restrictive an assumption in the case of free radicals, since the great majority of them have been found to fit into this category. In the absence of a magnetic field, these magnetic dipoles point at random, and hence all the unpaired electrons have the same energy E_0 (Fig. 1). A further assumption has been made in the foregoing statement: there is no coupling between the spins of neighboring molecules; or, in the language of physicists, there is no spin-spin interaction. This is again almost true in the case of free radicals produced by irradiation, since their dilution in the parent substance is a thousand-fold or more, and neighboring spins are then sufficiently far apart to involve no appreciable interaction. If a magnetic field is applied, the electron dipoles line up parallel and antiparallel to the magnetic field as a result of a competitive process between thermal motion (that tends to keep the orientation random) and magnetic interaction (that tends to line up all the dipoles parallel). The new energy levels will therefore be, as shown in Figure 1:

$$E_1 = E_0 + {}^1/_2 g\beta H \text{ for antiparallel alignment, and}$$

$$E_2 = E_0 - {}^1/_2 g\beta H \text{ for parallel alignment,}$$

where $\pm {}^1/_2$ stands for the quantized spin along the direction of the magnetic field, g is the Zeemann coupling factor (also known as Lande spectroscopic splitting factor or more simply as the g-factor), β is the Bohr magneton, and H the intensity of the magnetic field. The ratio of populations N_1 and N_2 of both levels is a function of the temperature and of the difference $E_1 - E_2$ according to the equation:

$$\frac{N_1}{N_2} = e^{-(E_1 - E_2)/kT}$$

where k is the Boltzmann constant and T the absolute temperature. Since the net magnetization of the sample is proportional to $N_1 - N_2$, it follows that it will increase with decreasing temperature and will tend to vanish at sufficiently high temperature. This is the behavior known as the Curie law of paramagnetic substances, whose first approximation is

$$\chi = C/T + \chi_0$$

where χ is the magnetic susceptibility per unit mass, χ_0 the diamagnetic contribution to the susceptibility, and C is a constant, characteristic of the substance, that depends on the number of unpaired electrons and their g-values. The value of g in the case of a free electron (paramagnetism due to spin only) is 2.0023.

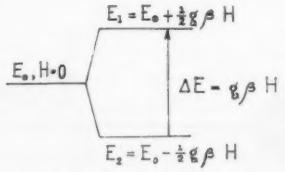

Figure 1. Levels of an unpaired electron in the presence of a magnetic field.

The small departure from 2.0000 is due to a quantum electrodynamical correction. The magnetic moment of the electron is, therefore, approximately β. However, larger departures from 2.0000 are observed, due to variable contributions of the orbital angular momentum of the electron to the total magnetic moment of the

This article is based on lectures delivered by the author for High School and College Teachers' Summer Institutes supported by the National Science Foundation, under the 1960 Visiting Foreign Staff Project.

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molecule. The knowledge of the actual value of the g-factor is therefore of main importance, this being one of the objectives of EPR. It should be noted that g is often anisotropic, i.e., its value depends on the orientation of the radical, and therefore is a tensor quantity. In these cases, measurements on single crystals are necessary. Returning to the scheme of levels shown in Figure 1, irradiation of the samples by photons of an energy precisely equal to the difference $E_1 - E_2$ will cause electrons in E_2 to absorb energy and be raised to level E_1. This is a typical process of resonance, since absorption of photons of either higher of lower energy cannot occur. The promotion of electrons from E_2 to E_1 is counterbalanced by their interaction with the lattice, through which they return to the lower level. This allows a steady absorption to take place. Measurement of the frequency ν of the photons and of the magnetic field H will suffice to determine the actual value of g, since the condition of resonance states

$$h\nu = E_1 - E_2 = g\beta H$$

where h is the Planck constant. Each value of g, therefore, determines the value of the ratio ν/H. Replacing the actual figures for the constants, one finds for example that using magnetic fields of the order of 3000 oersted, the frequency should be of the order of 9 kiloMegacycles/second for an approximate value of g equal to 2. This frequency corresponds to a wavelength of 3 cm (in the microwave region). Since microwave sources can be tuned only to within a very narrow range of frequencies, usually of the order of a few percent, it is much more satisfactory to work at constant frequency and make observations while varying H. The equipment for microwave production, developed for World War II radar, operated at 9 and 23 kMc/sec. Accordingly, most of the EPR results have been obtained at these frequencies. Commercially available equipments operate in 9 kMc/second (X-band). Lower frequencies, that could be obtained with simpler electronic circuits, are not always advisable because the signal-to-noise ratio decreases with decreasing frequency, and the consequent lower magnetic field would no longer be much higher than the magnetic fields due to surrounding nuclear magnetic moments, so that an electron spin would not line up in the direction of the applied magnetic field.

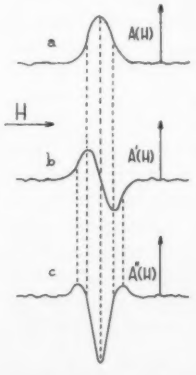

Figure 2. Curves of the absorption of microwave power, its first and second derivatives as a function of the magnetic field H.

Several types of equipment, as well as the typical operation, have been described elsewhere (*2*, *5*); the author refers the reader to the literature cited at the end of this article for further information on the subject. Essentially, the variation of the absorption of microwave energy by the sample as a function of the magnetic field is measured by conventional devices. Depending on how elaborate the equipment is, the measured effect can be either the absorption of energy or the variation of the absorption with time as a function of the magnetic field. Figure 2 shows typical curves in three cases.

Hyperfine Structure

In the foregoing, we have considered only the interaction of the electron with the applied magnetic field leading in general to a well defined frequency of resonance. Rather incidentally, when discussing the choice of lower frequencies, the magnetic field due to neighboring nuclei's magnetic moments was mentioned. Now we will discuss their influence in the observed spectra, which produces a further splitting of levels known as hyperfine splitting, and whose study is the second objective of EPR.

Let us consider first a nucleus of spin $^1/_2$. In the absence of a magnetic field, these spins (or magnetic dipoles) will point at random. If a magnetic field is applied, these nuclei will line up parallel and antiparallel to the magnetic field, just as in the case of the electron spins. However, since the difference between the nuclear magnetic energy levels is less by several orders of magnitude, the population of both states will not be affected by temperature to the same extent as in the case of electrons. At temperatures above a few thousandths of a degree Kelvin, therefore, the population of both nuclear levels will be, for practical purposes, equal. Consequently, if the unpaired electron is near one of these nuclei, because of the chemical composition of the molecule, for example, the probabilities will be equal for the electron experiencing a magnetic field higher than the applied one (due to the field of a parallel nucleus) or lower (due to an antiparallel one). Each electron level will therefore split into two, as shown in Figure 3*a*. Since the frequency of resonance of nuclei is very different, nuclei will not be promoted from one state to the other, so that permitted transitions of the electron will occur with constant orientation of the nuclei ($\Delta s = 0$). Accordingly, transitions simultaneously involving electron and nucleus are forbidden, and therefore not observed. Interaction with a nucleus of spin $^1/_2$, then, splits the spectrum of an unpaired electron into two lines (Fig. 3*a*). If the spin of the nucleus is 1, there will be three positions for the nuclei with the components of the spin, oriented with respect

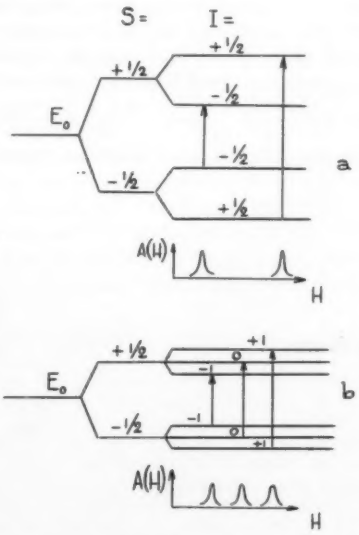

Figure 3. Interaction with a nucleus of (a) spin $^1/_2$, and (b) spin 1.

to the direction of the magnetic field, equal to 1, 0, and −1. As shown by Figure 3*b*, this leads to three lines in the spectrum since transition will be made only with $\Delta s = 0$. In general, if I is the spin of the nucleus, the hyperfine structure of the spectrum will show $(2I + 1)$ lines. Use is often made of this fact in the study of free radicals, since isotopic substitution of different spin nuclei of the same element often leads to information on the location of the unpaired electron in the molecule. Incidentally, this type of interaction, depending not only on the spin but also on the actual value of the nuclear magnetic moment, often permits an accurate evaluation of these characteristic constants. The hyperfine interaction is sometimes anisotropic, as is the g-tensor; in these cases, a study on single crystals should be made. Considerations of anisotropic interaction are, however, out of the scope of this article. In the following, we shall restrict our discussion to the cases of isotropic splitting.

Interaction with Several Nuclei; Spectra of Free Radicals

There is a tremendous variety of organic compounds of carbon and hydrogen, with and without oxygen. This latter element has zero magnetic moment (with the exception of ^{17}O which is very dilute in natural oxygen) so irradiation of organic compounds produces free radicals which have spectra whose hyperfine structure is due to interaction with protons ($I = {}^1/_2$). If interaction occurs with two equivalent protons, there will be four configurations for the nuclei;

$$(+{}^1/_2, +{}^1/_2);\ (+{}^1/_2, -{}^1/_2);\ (-{}^1/_2, +{}^1/_2);\ (-{}^1/_2, -{}^1/_2)$$

Of these four, the second and third are equivalent. There will actually be, then, three configurations, in the ratio of probability of occurrence 1:2:1, and the spectrum of such a species will exhibit three lines in the same ratio of intensities. Interaction with three protons will give four lines with intensity ratio 1:3:3:1, and so forth. In general, interaction with n protons will give rise to $(n + 1)$ lines, with a distribution of intensities determined by the coefficients of Newton's binomial. In saturated hydrocarbon radicals, the only protons that are equivalent are those bound to the element which also bears the unpaired electron and are near chemical neighbors. For example, irradiation of propanol would be expected to produce the two radicals:

$$CH_3—CH_2—CH_2 \quad \text{and} \quad CH_3—CH—CH_3$$

The former would produce a five-line spectrum, the latter an eight-line spectrum. The experimental evidence shows that a spectrum of eight lines in the ratio of intensities 1:7:21:35:35:21:7:1 is obtained. This suggests that irradiation produces as the more stable free radical, one having the unpaired electron in the middle carbon (*7*).

Studies on the rate of disappearance of the spectra at different temperatures also provide information in regard to the recombination and eventual appearance of secondary radicals. Studies made, for example, on ice, irradiated and measured at low temperatures, suggest the following reactions (*8*):

$$H_2O \rightsquigarrow H + OH \text{ by irradiation}$$

$$\left.\begin{array}{l} H + H \rightarrow H_2 \\ H + OH \rightarrow H_2O \end{array}\right] \text{ over } 20°K$$

$$\left.\begin{array}{l} OH + OH \rightarrow H_2O_2 \\ OH + H_2O_2 \rightarrow H_2O + HO_2 \end{array}\right] \text{ over } 100°K$$

$$HO_2 + HO_2 \rightarrow H_2O_2 + O_2 \text{ over } 130°K$$

In the case of free radicals, the results will, in general, aim at providing information on: location and magnetic parameters of the unpaired electron in the radical, identification of radicals, and kinetics of recombination.

It should be noted, however, that electron paramagnetic resonance is neither the sole research technique in this field nor the most important. Much information can be obtained by means of less involved techniques, such as optical, infrared, and ultraviolet spectroscopy and most of the common physico-chemical measurements. Electron paramagnetic resonance helps decide between species suggested by more readily available methods; it is essentially a high sensitivity technique.

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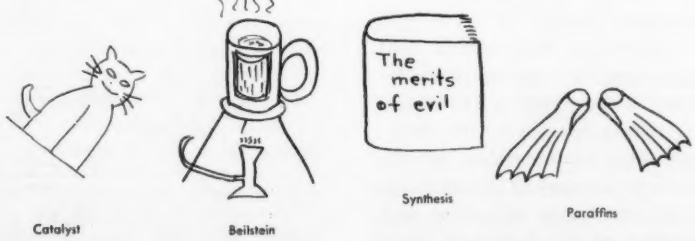
Chem Gems



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The Problem of the Sciences and the Humanities

A diagnosis and a prescription

A major cause of the developing cultural fission which I see as the problem of the sciences and the humanities is lack of effective communication between scientists and humanists. It is toward ways of improving communication, and bringing scientists and humanists more effectively together, that this essay is addressed. I present not only a diagnosis, but a prescription. This is a first step toward solution of the problem.

As scientists, we must be concerned with the fact that the products of our research are controlled and used far more by non-scientists than by scientists. Furthermore, it is through the arts that science must be interpreted to the layman who to some extent controls the products of our research through his votes and his dollars. He will not read the *Journal of the American Chemical Society* but he will read and be affected by the host of newspapers, magazines, and books which compete for his leisure time. It is a responsibility of the educated scientist to help in this interpretation, as it is the responsibility of the humanist to make the effort necessary for it, because otherwise an anti-intellectualism which begins with misunderstanding and fear can swallow up both science and art.

There is another extremely important reason for dealing in this place with this subject. It has to do with "speaking up." We are an educated part of our community, and it is our responsibility to have opinions on matters which affect us, our local community, and our nation. Many factors prevent us from fulfilling this responsibility. For example, many of our students are deficient in the ability to read perceptively, to write clearly, and to handle even simple mathematics. Our hopes to remedy this situation rest on those who recognize their plight enough to see that their children do not suffer the same disability. The degradation of our language which goes on apace via the sophistries of the advertiser, who dares not speak precisely and clearly because then his product is seen to be essentially like his competitors' (that it comes in pint bottles instead of the "big half-quart size") has left us subdued in the presence of questions, the answers to which require the most careful analysis and the most precise communication. And the cult of the expert, coupled with our own specializations which do indeed show us how difficult the problems of this world can be, leaves us unwilling to speak "out of our field."

This situation is dangerous for two reasons. One is that it is divisive: We are silent when we should speak—and silence can separate people as effectively as outright conflict. The other is that silence does not dispose of the problems. It merely creates a vacuum into which rush all manner of less inhibited "experts," often of doubtful repute, often with special axes to grind, often of extraordinary narrowness of knowledge and outlook.

I venture to offer a prescription which will help us to correct this situation. It will not be an easy prescription to follow because it involves the attainment in each of us of a reasonably balanced position with respect to the sciences and the humanities. Extreme positions are relatively easy to take. One merely fails to see all contrary evidences. Extreme positions are relatively easy to maintain; adversaries can come from only one direction. But the rational, balanced view is hard to take, because values must be weighed judicially. It is difficult to maintain because it will be attacked from all sides. Nevertheless, it has a certain nobility, rationality, and viability which must recommend it to us; and it gives us hope for the future.

Premises

Underlying every analysis and prescription there must be premises, whether implicit or explicit. It must be assumed, for example, that the problem contains elements amenable to analysis, and that an approach to a solution of it is desirable. Some of the premises such as these underlying my analysis are obvious; some must be stated explicitly.

I assume that scientists and humanists are concerned with the same world—even though they may look at different aspects of it and see it with different eyes. This is the basis for our image of the university, with its emphasis on wholeness and ideal lack of barriers between disciplines. I assume, then, that it is the same "whole" with which we are all concerned.

I assume that both art and science are universal in scope, in that there is nothing whatsoever which may not be a subject for artist or scientist. We observe, however, that they differ in emphasis. This is because there are some areas of knowledge and experience which

Based upon the lectures given as National Sigma Xi Lecturer, on the Southwestern Circuit, Spring of 1960. Based also, in part, upon a book to be published by Harper & Brothers in 1961.

Editor's note

The article here presented is a modified version of one originally published in The American Scientist, **48,** 383 (1960). *We are sure readers will agree with the Editors of* THIS JOURNAL *that few attacks on the problem specified by the title have been so thoughtfully conceived and attractively executed. Grateful acknowledgment is made to the Society of the Sigma Xi and the Board of Editors of* The American Scientist *for permission to reprint.*

are more amenable to the tools of one kind than of another. However, I must insist on universality of scope because I also insist that no one can predict a limit to the potentialities of human beings. Yet there does not seem to be any necessary reason why the works of the artist should be translatable into scientific terms, nor vice versa. There may be many different ways of speaking about the same things, and one way may be more fruitful than another, depending on circumstances.

I assume, in this connection, that too-great uniformity is undesirable, particularly in those areas where criteria of truth and validity are difficult to establish. This is why a universal language, if it could be achieved, would have certain disadvantages. It would be useful in much ordinary intercourse (and an approach to it is at present in existence in some areas of science) but it would be tragic if it displaced other languages. There are concepts which can be stated in one language which cannot easily be stated in another. As a general principle we must insist that variety has its uses: it allows flexibility, choice, and room for the appearance of the unpredictable.[1]

Such room and flexibility are essential because of a related assumption: *I assume* that no ideology or other dogma is capable of comprehending the infinite variety, invention, and new insight of which human beings are capable. This is why I speak of plural philosophies. One may extrapolate to some conceivable ultimate System, but it must be emphatically insisted upon that such an absolute is not attainable in fact. This is related to the success of liberal democracies in preserving individual freedoms of their citizens and in improving their lot and that of the world as contrasted to the totalitarian democracies which rest on dogma, or ideologies. These latter cannot afford to recognize phenomena which do not fit their dogma, and the insecurity which results when facts show that their dogma does not fit the world, reveals itself in tragic ways.

One more assumption, which probably does not exhaust all I will make, follows from the previous ones. It is that there cannot be any final solution to these problems—a solution which will usher in some state of Utopia. The passage of time brings new problems, some of which do not fit into the prevailing pattern.

Diagnosis

In order to offer an effective prescription I must make a diagnosis. This I will do by first offering two concrete examples which show how a poet and a physicist have communicated ideas of great importance. After discussing these briefly, I proceed to abstract from them. This leads us into a discussion of the structure of the university, and out of this arises the prescription in a natural way.

To deal with such complicated matters in a little space requires a directness of approach which will prevent me from citing minute detail and variety of opinion. Also, I have taken my concrete examples from a poet and a physicist since in a very real way they represent the clearest contrasts between humanist and scientist—much clearer, for example, than those between an historian and a psychologist. What I have to say, however, will be applicable to any humanist or scientist.

Two Pieces of Data

As our concrete examples, I have chosen a few lines from a longish poem "Einstein," written in 1929 by Archibald MacLeish,[2] and a formula from a work written 250 or more years ago by Sir Isaac Newton.[3] Both are taken out of context.

"... He lies upon his bed
Exerting on Arcturus and the Moon
Forces proportional inversely to
The squares of their remoteness
and conceives
The universe ...."

$$F \propto \frac{m_1 m_2}{s^2}$$

Now the first point that strikes us is the difference in appearance of the two. The poetry, written in English, can easily be read; the formula is obscure to anyone who has not studied physics. The formula says that there is a force (F) which is proportional to ($\propto$) the products of the masses of two objects (m_1, m_2) and inversely proportional to the square of the distance (s^2) between them. This formula may be rewritten for purposes of calculation as $F = Gm_1m_2/s^2$, where G is a constant of proportionality which adjusts the units used. There are certain limitations on this equation: it is approximated only if the distance s is *large* compared with the diameters of the objects, unless both objects are spherical. However, the application to the poetry is clear. Since m_1 and m_2 are not specified, *one* could refer to Einstein and the *other* to Arcturus, and since compared to their diameters the distance between them is large (it is about 32.6 light years) the formula would hold to a good approximation. Thus what the poetry says agrees, as a special example, with the formula.

Now, a good case could be made that no two people get exactly the same message from the poetry. This is because each important word is festooned with connotations, many of them quite different for different people. Thus the star Arcturus must surely carry different connotations for people from the Lone Star State, from those it carries for people from the other forty-nine. The connotations adhering to "moon" are known to change with the age and sex of the person.

On the other hand, it is very likely that all physical scientists understand essentially the same message from the formula as long as it is being used as a scientific communication. This is because the denotative symbols are essentially naked of connotations.

The formula may be used in another way, of course. The scientist may see it as a manifestation of the power of the human mind and imagination. He may ponder on the implications of Newton's creative insight. In this case he is not using it scientifically, but poetically. In this use it may *function* as a poem, or other work of art, though it does not have that *appearance*. In this use, different scientists will read different messages into the equation.

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[1] Read, J., J. CHEM. EDUC., **32**, 86 (1955).

[2] MACLEISH, A., "Collected Poems, 1917–52," Houghton-Mifflin Co., **1952**, p. 230.

[3] See, for example, HOLTON, G., "Introduction to Concepts and Theories in Physical Science," Addison-Wesley Press, **1952**, Chapter 11.

Another test I might apply to the two texts is that of translation from one language to another. If the previous statements carry conviction, then it must readily be agreed that the translated poem is unlikely to convey the translator's or the poet's thoughts exactly to his reader. The formula scarcely needs translation, since the symbols are part of an international scientific language, and the formula itself defines the force as gravitational. This partly explains the international character of science and the fact that nationalism feeds on the poetry, and particularly the songs, of a country.

To the poet the formula may seem cold, while his poem pulses with warmth and life. The scientist does not deny this. He is wise if he points out the price each pays for what he gets. By paying the price of lack of warmth, the scientist achieves wide communicability of a very precise and quantitative message. The poet achieves his warmth, but communicates a quite private message to whoever can hear. It is largely a matter of emphasis, of course, since the way the world is constituted allows us to *attain* no absolutes. The poem has its denotation, but this is the least of its messages; the formula has its connotations but these are essentially excluded in its scientific use.

Tools for Communication

The question may be raised: what kind of truth does each communicate? A good approach to an answer has been given by John Hospers.[4] He characterizes the truths of science as "truth-about" the objective world. This is a kind of truth which is publicly verifiable. Anyone with the requisite training can check Newton's formula to a reasonable approximation by carrying out one of the famous experiments or calculations which support it. Or, he can devise a different, hitherto untried, experiment and, provided he meets the limitations of the formula, he will confirm it. The formula says, in effect, that this is the way certain aspects of the world (masses of objects, distances between them) are related to each other.

The arts communicate truth of an essentially more private nature, "truth-to." This is a kind of truth which is validated by the communicant out of his own experience. The composer, artist, or author produces a work which, as John Dewey has said, creates participation of the listener, viewer, or reader in the artist's experience. Perhaps the artist has created a character who is recognized by his readers as "truer to life than life itself." Thus out of his own experience, or some felt need which is satisfied by the work, comes a personal validation of the work as true.

These differences between art and science are closely linked to the tools for communication used by artist and scientist.

Ratio. In science the drive always is in the direction of quantitative statements, the goal being relationships that can be stated by means of equations or formulas of the type of the example. This equation states a *ratio.* As such it reaches the acme of precision. The constant G is present to rationalize the units of s and m. Given G and the values of any three of the four quantities, the fourth can be calculated with complete *mathematical* certainty. Any uncertainty resides in the values of the quantities themselves. The ratio (or formula) is thus a very powerful mathematical tool.

Analogy. The majority of relationships which are studied by scientists cannot be stated in terms of ratios of the type illustrated. They can be put into mathematical form, but the equations contain adjustable parameters. Now the other important tool of the scientist is *analogy.* An analogy is a relationship of likeness, as is the ratio, but it is an imperfect ratio. In a sense, it may be likened to an equation with many adjustable parameters. Such an equation may allow a number of different theoretical interpretations, and one problem of research is to find out which, if any, of these is best—the criteria of "best" being correspondence to experiment, and fruitfulness or correct predictions (among other criteria). Analogy is the principal tool for communication of relationships (as distinct from mere description of phenomena) in the biological, social, and related sciences. But the drive is always toward quantitative statement.

Metaphor. In the arts, too, one of the principal tools of communication is analogy. Yet there are many things which cannot be communicated even by this tool. These are the things which verge upon the ineffable. They must be communicated and, when in our need we feel that we must, we turn to the greatest artists who have from time to time succeeded in this nearly impossible task. They have invoked the aid of *metaphor.* We quote them, or point to their work, and thus substitute their genius for our fumbling. Metaphor is the most powerful tool of the humanist. "Its efficacy verges on magic," says Ortega, "and it seems a tool for creation which God forgot inside one of His creatures when He made him."[5] By the aid of metaphor it is possible to attain a qualitative precision which is *functionally* analogous to the quantitative precision achieved by the use of ratio.

The emphasis in science is on ratio and analogy, whereas that in the humanities is on metaphor and analogy. But the humanist does not hesitate to approach the use of ratios—as in stylometric analysis—if it suits his purposes; and the scientist now-a-days is sufficiently secure that he can turn to metaphor, as when the nuclear physicist speaks of "magic numbers," not knowing, at first, what they signify.

The Whole University

A university catalogue lists, already defined in it the various disciplines that are taught. If there is doubt about any one of them, one can go and ask the people who are members of the particular department, what they do. Or, one can observe what they teach, and what they publish. I shall group departments: physical sciences include physics, chemistry, geology, astronomy; life sciences include botany, microbiology, zoology; on the border between physical sciences and life sciences would fall biochemistry, biophysics, ecology; and so on. This grouping is done for convenience of representation, and because straight enumeration will not accomplish my purpose, since the individual disciplines cannot be arranged in an entirely

[4] Hospers, J., "Meaning and Truth in the Arts," University of North Carolina Press, 1946.

[5] Ortega y Gasset, José, "The Dehumanization of Art and Notes on the Novel." Tr. by Helene Weyl, Princeton University Press, 1948, p. 33.

logical sequence; they did not develop in that way. So, by grouping them I can avoid trying to make minute distinctions while preserving the larger differences.

I then proceed to define the Sciences and the Arts (or, Humanities) in the following way. I arrange these groups in a logical sequence around the perimeter of a circle, as is shown in Figure 1.[6,7] The two arcs opposite each other comprise the Arts and Sciences, as labeled in the figure. It can be seen that a certain internal logic is displayed by the figure. On the Science

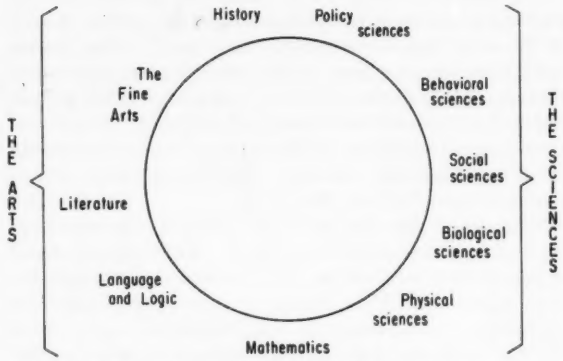

Figure 1. Definition of the Arts (the Humanities) and the Sciences.

side, there is (by and large) a sequence from inorganic to organic; from preoccupation with statistical aggregates and chance events to concern with phenomena displayed by individuals, and small groups of individuals, and events controlled by individuals and small groups. On the Arts side there is a sequence from discursive to presentational means of communication, with an emphasis at the upper part of the arc on the more local or nationalistic expressions, which verge then into History.

I explicitly do *not* wish this figure to be taken too literally. Disciplines across from each other are not opposite, or opposed in any real way, nor is the width of the sectors given to a discipline meant to imply anything but typographical convenience. What I do explicitly imply is that the whole represents a connected *field*, without natural barriers; that in defining the Arts and Sciences in this way I do convey a kind of oppositeness between them in the large, in that there are areas of knowledge and experience which in actual fact each takes as its own; and that there are disciplines which belong to both areas: Mathematics, one of the oldest of the humanities and newest of the sciences, and History, which finds itself sometimes under the Dean of the Humanities, sometimes under the Dean of the Social Sciences.

In *all* disciplines of the university which have intellectual content there are practiced three kinds of activities: analysis, synthesis, and reduction to practice.

Analysis is the activity of gathering data, describing things as they are, collecting and recording instances, making lists, and so on. Now an intelligent person cannot go very far in this kind of activity before he begins to see patterns in his data, likenesses in his descriptions, similarities among the separate instances. As this occurs, the essentially analytical activity goes over into a synthetic one.

Synthesis is the activity of finding connections between the data made available by the analytical activity, making hypotheses and theories, and developing laws. In short, synthetic activity is a generalizing activity. It involves abstraction, for the general statement is at a higher level of abstraction than the many single instances on which it is based. But the hypothesis, theory, or law must be reduced to practice.

Reduction to practice is the activity through which a return is made from the general principle to the single instance. It may be carried out to see if a predicted fit is obtained. It is then the experiment designed to test a theory. It may be the examination of a document to see whether it meets some critical test. It is also the creative activity by which the work of art or science is produced.

There are two features of these activities which I must emphasize. One is that it is difficult in many instances to classify some activity as clearly of one or the other kind. I have had to separate them for the purposes of definition. But when we try to apply our concepts to actual cases we find that we are dealing with a mixed situation, and we have to recognize not pure analysis or pure synthesis, but *emphasis* more in one direction or the other.

The second feature is that these activities may be carried out at many levels of abstraction. Thus theories may be the subject of analysis, which might then lead to hypotheses or theories about *them*, and these higher-level generalizations may be tested by reduction to practice. Or, methods of reduction to practice may be collected and examined with a view to the heuristic enterprise of developing the method of the methods, that is, theory at a more abstract level.

Sphere of Knowledge and Experience

The foregoing discussion and definitions permit me to illustrate in a manner which must not be taken as more than an analogy, a concept of the structure of the university. This is shown in Figure 2. The figure is idealized as a sphere, not because the ancient Greeks considered this to be the most perfect figure in solid geometry, but because it helps to avoid invidious comparisons. The circle of Figure 1 which I used to define the Arts and Sciences now comprises the equatorial belt of our sphere. Toward one pole I gather the technologies; toward the other the philosophies.

The technologies I define as disciplines wherein reduction to practice is emphasized. But I must insist that, like any other university discipline with intellectual content, the technologies comprise analysis and synthesis, and as a part of the university they comprise the heuristic activity I described above: the analysis and generalization at higher levels, of the technology. If pure reduction to practice were involved, we would not have a technology, but a craft. In this definition I do not derogate the craft, but distinguish it clearly from the technology.

[6] An early form of this diagram was first published by CASSIDY, H. G., J. CHEM. EDUC., **32**, 86 (1955).

[7] See also, FULLER, E. C., J. CHEM. EDUC., **34**, 110 (1957).

Every discipline in the Arts and Sciences has its conjugate technology. In the sciences these are often called engineering; in the arts they have special names. And it is a characteristic of the technology that it often calls upon many disciplines, which it then fruitfully combines. Thus medicine is the technology of a whole constellation of disciplines, ranging from physics and chemistry to literature and drama. Most kinds of engineering include mathematics, physics, and a number of other sciences. Law is the technology of jurisprudence, and calls upon anthropology, psychology, and much more. The technological disciplines of literature and the fine arts—the "commercial" disciplines—as also drama, call upon many kinds of knowledge and experience. I do not need to enumerate all the technologies. They can be found in university catalogues, and are recognizable via our definition.

The philosophies I define analogously to the technologies. In them synthesis is emphasized. Here, too, a number of disciplines are likely to be drawn together in fruitful union. Here, too, I emphasize plurality. This is because the idiom appropriate to a philosophy of science cannot be transferred to a philosophy of art with any real meaningfulness—at least at present. We can understand this difficulty on the basis of our examination of the tools of communication: metaphor has its uses, and they are not the uses of ratio. Moreover, we do not find notable agreement among philosophers, and so we are led to the conclusion that each discipline or group of disciplines should have its philosophers, who in general should first, or concomitantly, be experts in that area.

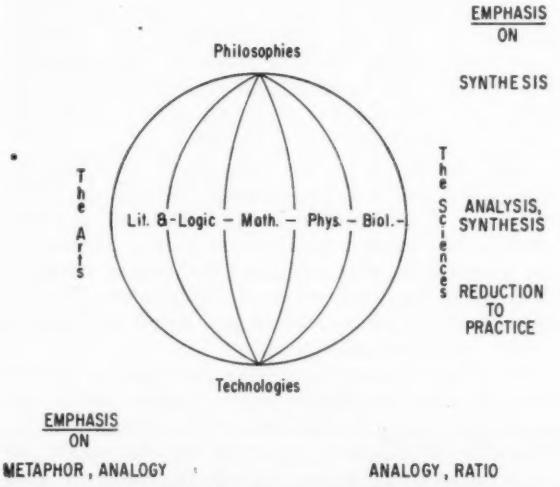

Figure 2. Conceptual analysis of the structure of the university. The equatorial band of disciplines, shown in part, is taken from Figure 1, and extends around the sphere of this figure.

What I wish explicitly to emphasize is that the sphere of knowledge and experience comprised by the university is a *whole*, in principle, without barriers. I suggest also that, reading downward, synthetic activities are emphasized near the "top" part, analytic and synthetic near the equator, and reduction to practice near the lower part. *All three are found throughout* but the *emphases* differ in the different classes of disciplines. In the Arts hemisphere, to repeat, analogy and metaphor, whether discursive or presentational in their utterance, are the chief tools of communication; in the sciences, analogy and ratio.

This figure gives us an idealized picture of the intellectual structure of the university. A college would be pictured in much the same way, except that fewer disciplines would be represented, and many of the technologies and philosophies would be absent. A technological institute might comprise mostly the lower half of the diagram, or in other cases might be indistinguishable from a university.

I have, of course, left out of consideration a great many aspects of the arts and sciences. But this essay is not meant to be exhaustive. Its chief function is to analyze the particular situation as I see it, and present this as a starting point for a suggested prescription. A great deal has been written on the subject of the cultural cleavage between humanists and scientists.[9] Most of it, however, describes the situation and deplores it, without suggesting remedies. I grant that remedies for such a complicated group of illnesses are hard to come by, but I will proceed nevertheless to offer a prescription based on the foregoing analysis, since it is part of my argument that we must speak up, and attack this dangerous situation.

Prescription

In the preceding sections of this essay I have provided the means for constructing a prescription which now naturally follows. It is a prescription for the treatment of underlying causes. The important point is that this prescription, to be effective, must be applied by *individuals*, and this means intelligent people who recognize the problems, and are willing to accept the responsibility to speak out, and to set an example. It is not a legal prescription. We have the laws, but this is prior to written law.

Mutual Understanding and Criteria for Valuation

The prescription contains a number of ingredients. One has to do with mutual understanding, which is a prerequisite to mutual trust. Another has to do with fortification of a point of view which supports our actions and which guides them through rational criteria of value. Still another has to do with means for applying the prescription.

We have seen that all of our intellectual endeavors are in principle understandable and capable of being connected to each other. If the scientist sees the artist depending heavily upon metaphor, that tool capable of such qualitative precision in skilled hands, he surely should be able to understand. He knows from his own work, and his human reactions to it, that there are aspects of experience which cannot be stated in quantitative terms. Even *he* uses metaphor when he is faced with the need to communicate such

[8] The meanings of these diagrams are more fully discussed in Cassidy, H. G., "The Sciences and The Humanities," Harper & Brothers, 1961.

[9] Forbes, W. F., and Story, G. M., J. Chem. Educ., **34**, 594 (1957). See also Moberly, Sir Walter, "The Crisis in the University," S. C. M. Press, 1949; Snow, Sir Charles P., "The Two Cultures and The Scientific Revolution," Cambridge University Press, 1959; Ashby, Sir Eric, "Technology and the Academics: an Essay on Universities and the Scientific Revolution," The Macmillan Co., 1959.

experience. The skilled worker, recognizing skill in another area can afford to praise it. At the same time, the humanist, if he were able truly to recognize the power of a scientific statement, and the quantitative accuracy with which an idea can be communicated to *anyone* who can listen, can surely understand that here is a manifestation of interaction between human beings worthy also of praise.

As a class exercise, in a course in elementary physical science which I teach, I once asked a group of Yale freshmen to interpret the few lines of Mr. MacLeish's poem which were quoted above. These men had been studying Newton, and all of them saw that here, on the face of the verse, was a statement of a special case in which Newton's Law of Gravitation held, as the Poet implied. But several of them (about ten percent) went further. They said that this was only a minor point. What the Poet says is that Einstein exerted two forces. One of them, the gravitational force, is small—in effect trivial. The other, truly powerful force, was the conceptual one, through which, in "conceiving the universe," he changed our entire way of thinking.

These same students could also be impressed with the tremendous scope and quantitative precision of Newton's gravitational formula. Certainly the gravitational force exerted by Einstein's body upon Arcturus is negligibly small, yet it is comprehended by the gravitational formula. It seems to me, then, that when scientists and humanists can see these powers in each other (and the limitations to these powers), the precondition exists for mutual understanding.

Scientists and humanists (artists) are connected, too, by the fact that both make use of analysis, synthesis, and reduction to practice as they go about their work. This particular connection is a vital one because in it lie the means for deeper understanding of each by the other, and especially because in it lie the means for evaluating the work of each by the other.

To be able to see that both humanists and scientists rely upon analysis, synthesis, and reduction to practice, as functions, though the results are very unlike in appearance, gives further ground for understanding. But the soundest basis lies in the realm of valuation and a point of view.

The greatest value inheres in that work which brings together constructively, in the sense of conjoined analysis, synthesis, and reduction to practice, the largest area of truth and meaning. This applies to the public "truth-about" of the scientist as well as the private "truth-to" of the artist's communication. It applies to the more denotative meanings of the scientific statement as well as the more connotatively rich meanings of the humanist. One of our criteria of value then, is that *wholeness and large compass, with depth, count for more—and much more—than partialness and superficiality or narrowness.*

Another criterion of value is part of this one. It is that *rationality is superior to irrationality*, for, as Howard Mumford Jones has pointed out, it takes rationality to discover irrationality, not the other way around.[10]

Why do I argue that wholeness and large compass with depth in any work make it more valuable than the lack of these? It is because when I see many interconnections between the parts of a system I feel more confidence in the validity of the system than when there are few. A theory in science which reaches from the atoms to the stars with reasonable fit to the data of such disparate realms gives us confidence that it carries truth which is not given by a theory which fits a few local instances. A theory of forms in art which can deal with art over a span of two thousand years gives us more confidence than a pronouncement, no matter ow sonorous, about some local revolt in art.

And why do I praise rationality and meaningfulness over irrationality and meaninglessness? It is because the human race has found the former hard to come by. It requires effort in the same sense that staying alive requires effort. It is part of the grand pattern in which we see living beings developing in constant struggle against the degradative forces that lead to death; in which we see human beings lifting themselves, ever so slowly, and with what untold effort, above the chances, accidents, and irrationality of their animal origins.

The application of these criteria of value helps to explain or clarify many phenomena in the work of humanists and scientists. Perhaps we have felt dismayed by the shallow, even ugly, nature of much literature, painting, music, of the last several generations. Of course the contemporary often suffers in contrast with the past. The dross of the past has largely perished, or lies gathering dust in a museum or archive, which amounts to the same thing; and the best remains, against which to test the new. Also the contemporary is likely to depart from past patterns because the artist (as, too, the scientist) must keep seeking for new ways of communicating what comes from his continual reaching out into the unknown. This is not what I refer to, but to shallowness and ugliness intrinsic to the work.

Early science is always analytical. You have to collect data, and describe and report phenomena, before you begin to see connections between them, and patterns in the whole. A Linnaeus and a Mendeleev, the generalizers who recognize the largest patterns, come late in the development of any science. This is a state of affairs which leads many people to think of science as entirely analytical.

Out of such misunderstanding has come a spate of literature over the last seventy or eighty years: the reportorial, the minutely and solely descriptive, the stream-of-consciousness types of writing which are surely unsatisfying as art, though they may be good reporting. They are unsatisfying, and misguided, on three counts. One is that they attempt to be scientific but imitate *partial* (analytical) science. Another is that they try to imitate science at all. We have seen that science and art are quite distinguishable in terms of their emphases on means of communication. The artist who attempts to imitate science runs the grave risk of losing his integrity as an artist. For if he were really successful, he would become a scientist, and no longer an artist. If he fails, then he has abdicated his duty to no valuable end. The third cause of failure is that they are largely analytical. This is why they are unsatisfying, and ugly. The

[10] JONES, H. M., *Harvard Educational Review*, **22**, 203 (1952), pp. 209–210.

great tradition of humanism confirms that humanists are concerned with wholes. No wonder reporting, by itself, seems superficial when masquerading as literature; why painting which is imitative without understanding and a theory is ugly.

Ignorance of a similar kind is found in technology, philosophy, and science. For example, it may be discovered that a certain spray will be extremely effective in killing mosquitoes, or their larvae. Wholesale spraying, then, of parks, fields, streams, does indeed kill off the mosquitoes, but also the birds and fish which, had they been fostered, might have done a reasonably good job in the first place. This kind of error lies in mistaking analysis for all there is, or in looking at a complicated system with a very low-level-of-abstraction viewpoint, narrowly restricted. The proper functioning of analysis, synthesis, and reduction to practice would lead one to think of the whole situation. This is what ecologists are trained to do, and a well-trained ecologist would not commit such mistakes.

Application

These examples serve to suggest how our criteria of value may be applied. We ask of a work, whether of art or science or philosophy or technology, "does it fruitfully unite analysis, synthesis, and reduction to practice?" If it does, then it is greater and more valuable than a work which does not. This is not to deny value to the latter works. But we need to be able to compare, with reasonable assurance, the communications with which we are continually faced. We can then surely say that some potboiling experiment, or the collection of a series of properties of compounds, though having merit if well done, is less valuable than work containing elegant reasoning, or one which derives a relationship. We can look at a canvas on which has been splashed some handfuls of paint, and agree that it has some local merit as expressing the painter in his honest effort to devise an effective idiom, perhaps, but we would not rank it the equal of a canvas which carries to a group of artists a dream, or message of hope, power, or enlightenment.

What we are saying is that the application of these criteria, which are derived from a consideration of the whole compass of knowledge and experience as it is manifested in a university, enable us to evaluate in a reasonable way a great many of the utterances of our culture, whether they be works of the mind or the hand. Moreover, such evaluation can be objective and not imply condemnation of a work because it has merit only, and not great value. But we can demand of great art, and of great science, not only that it show us the way things are in this world—a good reporter can do that analytical job—but also that it show us how things could be. We ask that it elevate the spirit; that it lift us out of ourselves; that it set us an example of the heights to which the human being can ascend; that it show us how to ascend these heights.

Resume

I have endeavored in this essay to derive a few of the general principles which are common to, and so connect and unite, the arts and the sciences. I have shown that there are authentic differences between science and art in terms of the means of communication used and the kinds of data amenable to the tools of artist and scientist. At the same time, I showed that the artist and scientist use the same conceptual tools, analysis, synthesis, and reduction to practice, in performing their labors, and thus they meet at this common intellectual level. Here they are able, if they will look at *function*, to see these tools in operation, and not to be misled by the myriad differences in appearance of the results.

At the same time we are enabled to evaluate the works of the artist and scientist: that work in which the combined use of these tools produces a work which elevates the human spirit and shows us of what noble thoughts and acts we are capable, is greater than one which does not, or does less. Thus we have criteria, even in areas outside our specialties, for forming responsible opinions.

New Titles in Nuclear Science Series

A new AEC-sponsored series of monographs, Radiochemical Techniques, is beginning to appear, parallel to the series on the radiochemistry of the elements. The same general style is used, including a general review of the technique, a discussion of principles, and a detailed survey of applications; a collection of selected procedures which use the technique has been made from the literature. The object of the series is to cover techniques not reviewed elsewhere. As with the other series, the Subcommittee on Radiochemistry, National Academy of Sciences—National Research Council, plans periodic revisions as new information develops. The cooperation of readers in assembling this information is requested. The NAS-NS volumes are available from the Office of Technical Services, Dept. of Commerce, Washington 25, D. C.

3101 Liquid-Liquid Extraction with High-molecular-weight Amines (Fletcher L. Moore), $1.00.

3102 Separations by Solvent Extraction with Tri-*n*-octylphosphine Oxide (J. C. White and W. J. Ross), $0.75.

3103 Low-level Radiochemical Separations (T. T. Sugihara), $0.50.

The AEC-sponsored series, Radiochemistry of the Elements, now numbers 36 volumes, NAS-NS 3001–3036. [See THIS JOURNAL, **37,** 386 (1960); **38,** 72, 349 (1961).] Each is a monograph containing a general review of the pertinent chemistry and compiles the latest (1960) radiochemical information, and is available from the address given above.

3032 Aluminum and Gallium (John E. Lewis), $0.50.

3033 Antimony (William J. Maeck), $0.50.

3034 Titanium (Chong Kuk Kim), $0.50.

3035 Cesium (H. L. Finston and M. T. Kinsley), $0.75.

3036 Gold (J. F. Emery and G. W. Leddicotte), $0.50.

William E. Morrell
National Science Foundation
Washington, D. C.

Review and future plans

Summer Institutes

The National Science Foundation supported its first summer institutes for science and mathematics teachers in 1953. However, similar programs had been sponsored earlier by other agencies. For example, General Electric institutes for high school teachers date back to 1945. Oklahoma A. and M. College and the ACS Division of Chemical Education Committee on the Teaching of Chemistry, under the vigorous leadership of Dr. Otto M. Smith, co-sponsored a 10-day "Workshop" at Stillwater in 1950 for college chemistry teachers. Less than $2000 was available for operating costs, and participants paid their own expenses or wangled train fare from their college presidents, but the 10-day exchange of ideas proved so stimulating that similar sessions were organized again in 1951 and in 1952. Then it was felt that these meetings should be extended to other campuses, so Dr. Smith's committee obtained the cooperation of the California Institute of Technology and Pennsylvania State University as host institutions for 1953, followed by Kenyon College, North Carolina State College, and the University of Wyoming for 1954.

In 1953 the NSF supported its first summer institutes, one for college teachers of mathematics at Colorado, and one for college teachers of physics at Minnesota. In 1954 the Foundation made $10,500 available to the University of Wyoming to lengthen the already-scheduled Stillwater-type "Workshop" to five weeks. The Division of Chemical Education Committee on Teaching planned the program, believing that the movement might really spread if the Laramie meeting could be made an outstanding success. It sought the cooperation of leaders in chemical education. The response was more than enthusiastic. However, one who agreed to give a series of lectures attached a qualification: he would participate only if the name "Workshop" were abandoned. Thus the first Chemistry Institute was born, co-sponsored by the National Science Foundation, the University of Wyoming, and the Division of Chemical Education. Its theme was Recent Advances in Chemistry.[1]

A simultaneous institute for high school chemistry teachers was organized, to operate in parallel with the institute for college teachers, and with joint sessions when appropriate. The Ford Foundation Fund for the Advancement of Education provided $15,000 for the institute for high school teachers; 56 high school chemistry teachers each received an expense grant of $150.

These chemistry institutes were carefully observed by National Science Foundation personnel, and exerted considerable influence on later developments.

In that same year, 1954, the NSF supported three other summer institutes, two for college teachers of mathematics and one for high school mathematics teachers.

At a meeting in late 1954 of officers of three ACS units concerned with chemical education, the Division of Chemical Education, the Council Committee on Chemical Education, and the ACS Board Committee on Education and Students, the remarkable stimulation that resulted from the "Stillwater Workshops" and the Laramie institutes was discussed. One of those present, a representative of industry, mused: "Just think of the benefits that could result if such meetings could be made available to 1000 chemistry teachers!" Making some quick calculations, he estimated that the cost might be $1,000,000, but then he urged earnestly that the idea not be abandoned without serious investigation. He felt that the money might be obtained from chemical industries. Of course he was appointed chairman of a fund-raising committee, with the hearty approval of his academic colleagues who had worked so hard to raise the $1500 spent by an entire Stillwater-type "Workshop." In later years chemists who participated in that discussion have been heard to reminisce: "Do you remember when we wished we had $1,000,000?" The $1,000,000 which seemed so visionary at that time did become available, but in a manner not then anticipated.

The NSF had provided funds for two institutes in 1953, four in 1954, and then the number grew to 11 in 1955, followed by 25 in 1956. In the meantime, congressmen had learned of the effectiveness of the institutes, and suggested that the Foundation's budget request for funds for education in the sciences be increased. Consequently, for 1957 it was possible to schedule 96 institutes, but most of these were financed from money which was earmarked by Congress to be used "in connection with a program of supplementary training for secondary school science and mathematics teachers." This limitation differs from most budget limitations in that it sets a minimum rather than a maximum that shall be devoted to a particular objective. It specifies a floor rather than a ceiling with respect to the amount that shall be utilized for high school teachers. Each subsequent appropriation to the Foundation has included a corresponding limitation or "floor."

The Foundation supported 126 summer institutes in 1958. An effect of Sputnik was apparent in the financing of 350 institutes in 1959, followed by 381 in 1960, and 398 in 1961.

A typical NSF summer institute provides stipends and travel and dependency allowances for about 50

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[1] See THIS JOURNAL, **32**, 262 (1955).

teachers. The stipend from NSF funds may not exceed $75 per week. The maximum dependency allowance is $15 per week per dependent up to four. The maximum travel allowance is $80, at not more than 4¢ per mile for one round trip between home and institute. Most of the institutes are six or eight weeks in length. The average grant from the Foundation for a summer institute for high school teachers is about $58,000, of which slightly more than one quarter, on the average, goes to pay the operational costs. In the NSF summer institutes for high school teachers, $43 is the average operating cost per participant per week.

The program in the institute is planned and conducted by the host institution, usually a degree-granting college or university; the participants are chosen and stipends are awarded by the host institution. Some institutes are designed for teachers who have a very weak background in the subject matter, others are planned for teachers whose backgrounds are stronger. Many institutes and participants have recommended that as homogeneous a group as possible be selected by any particular institute, and conversely, many have reported that the greatest difficulties in institutes have arisen through enrolling heterogeneous groups. Specifically suitable courses can be designed and the courses are much easier to teach when the participants have similar backgrounds, and participants with similar backgrounds but who come from a wide geographic area are likely to learn more from each other than participants whose backgrounds differ widely or participants from a single region.

The stimulating benefits that participants gain from each other, are frequently reported to be as great as the benefits from the classroom. The benefits from group interaction constitute one of the principal advantages of institutes over ordinary summer schools. Many institutes "set the stage" so that participants will have maximum opportunity to learn from each other. For example, they house participants in an area reserved for the institute; they schedule the group to eat together at least once each day; they provide lounges where impromptu discussions can occur at the drop of a hat or the drop of a provocative phrase. In a well designed and stimulating institute, learning goes on almost every minute the participants are awake. Commuters miss so many of the plus values of an institute that in some institutes stipends are given only to individuals who will live with the institute group.

The 398 summer institutes scheduled for 1961 will provide stipends for about 18,000 high school teachers and 1800 college teachers of science and mathematics, at a cost of about $22,700,000 to the NSF.

Thus has the wistful wish of only a few years ago come true, the wish for $1,000,000 to provide summer refresher training for 1000 teachers. It has come true beyond the wildest dreams then imagined. In the phenomenal growth of the institute program, those "Workshops" of the Stillwater type, sponsored some ten years ago by a committee of the Division of Chemical Education, have had enormous influence. For example, every person who has held the title of "Program Director for Summer Institutes" at the National Science Foundation (J. Arthur Campbell, Grant W. Smith, Edward L. Haenisch, and the writer) gained experience through active participation in those "Workshops."

Summer Institutes for elementary school personnel have been sponsored by the Foundation since 1959. This program is classed as experimental, pending study of the enormous problems involved in improving the teaching of science and mathematics in the elementary schools. The magnitude of the task is hinted at when one recalls that there are approximately 1,000,000 elementary school teachers in the nation. A very large number of these have little background in either science or mathematics. Typically they dislike these subjects and feel insecure in teaching them. Solutions are being sought, but it seems apparent that those who teach science and mathematics in the elementary schools should understand the basic principles of science and mathematics. The role that the National Science Foundation should play in this area has not yet been determined. In the meantime, the Foundation has provided funds for 19 Summer Institutes for Elementary School Personnel in 1961. Less than 0.1% of the elementary school teachers and supervisors will be able to participate.

The summer institutes sponsored by the NSF have been from four to twelve weeks long. But many participants in the Stillwater "Workshops" would argue that there is still a place for the shorter meetings for college teachers, meetings from several days to a few weeks in length. Many college teachers must teach in summer schools and summer institutes; for these and other reasons they are unable to attend summer institutes themselves. But they would welcome the stimulation of shorter meetings, with attendant opportunities to discuss recent advances in science. While the NSF institute program was mushrooming, a committee of the Division of Chemical Education continued to seek support for meetings of the Stillwater type. Harry Lewis, the chairman of this symposium, was especially active and successful in obtaining finances from industry, particularly from paper companies, to support several "conferences" for chemistry teachers. Finally, in 1959, considerably influenced by the chemists, the National Science Foundation began programmatic support of Summer Conferences for college teachers. ("Summer Conferences" differ from "Summer Institutes" in being less than four weeks in length.) A Summer Conference typically considers subject matter in a limited field. The NSF is supporting 23 conferences in 1961 in the Summer Conference program as such, but it also supports individual conferences of many kinds, varying from exploratory discussions of curriculum revisions to high level symposia for specialists.

A Look to the Future

What should be the future of institutes for science teachers? Two major questions must be answered before this can be decided. (1) Have the institutes been effective? (2) Have they completed their task?

Have the institutes been effective? The vast majority of comments that reach the Foundation, from institute participants, directors, lecturers, and visitors, indicate strong conviction that most of the institutes are very

successful and effective, more effective than could be anticipated when they were first tried. Objective evidence, however, is very difficult to obtain. Many local studies have been undertaken, and in general they confirm the belief that the institutes have been successful. Studies have also been made on a national scale. The most thorough of these investigations was conducted by an independent private agency, the Bureau of Social Science Research, Inc. In earlier studies this organization sought to determine the effects of the 1957 institutes on high school teachers who participated. These same teachers were studied again in 1960, in an attempt to evaluate persistent effects of the institutes. The teachers themselves were interviewed, as were their colleagues, principals, and supervisors. Efforts were made to identify changes in their teaching attributable to institute participation, and to compare the effectiveness of these teachers with that of similar teachers who had not attended institutes. A 600-page report of this study has recently been received by the Foundation. Summaries are being prepared and will be made available for distribution. In the study, approximately 1500 persons were interviewed. The conclusions, which will be fully reported at another time, confirm earlier indications that the long-range, as well as the immediate, effects of the summer institutes are more than gratifying.

Have the institutes completed their task? Far more has been spent than the $1,000,000 that was wished for at an earlier time, and the institutes have been more effective than was anticipated. But the job is far from finished.

Should the institutes program be converted to a program of fellowships? If fellowships were substituted, in numbers and amounts equal to institute subventions, total cost would be lowered somewhat since presumably tuitions would be paid in place of total operational costs as in the institutes. The resulting saving would probably be about 10%, but at the expense of probable loss of two of the most important and helpful characteristics of outstanding institutes: courses specifically suitable to the needs of teachers (suitable in pacing as well as content), and benefits that participant-teachers derive from a homogeneous group, stimulating and learning from each other. Replacing institutes by a large program of fellowships appears less likely now than was expected.

A problem that is likely to become increasingly troublesome is that of repeated participation in institutes. The Bureau of Social Science Research study referred to above presents strong evidence that participants benefit more from two institutes than from one, and from three more than from two. At present, however, there is insufficient evidence to answer the question: In general does the nation benefit more if one teacher participates in several institutes or if several teachers each participate in one institute, at the same total cost? Which is the more advantageous expenditure of the available money? At present we believe that there is no general answer. The NSF "Guide for 1961 Summer Institute Directors" states:

> If a teacher is working toward a specific goal, such as a Master's degree in a coordinated sequence, stipends in several successive summers might well be justified. But it is doubtful whether stipends should be given in successive summers, in either the same or different institutes, to teachers who have no well defined plan for coordinated study toward a definite goal.

It seems apparent that teachers who have not yet received institute stipends should receive preference over those whose repeated attendance has been supported to the point of diminishing returns. Many teachers who most badly need the help of institutes have not yet even applied; others have been turned down repeatedly.

Evaluation and selection of applicants is a problem likely never to be solved to complete satisfaction. A parallel problem is the evaluation and selection of institutes to receive or retain financial support. One aspect of this problem is that if support could be assured for several years at a time, planning by host institutions could be greatly facilitated, and long-range planning could be on a much more rational basis.

In the rapid evolution of the NSF Summer Institutes program, changes are occurring which, it is hoped, may be partial solutions to difficulties mentioned above. One of these developments is the specific recognition of institutes of sequential type. These may involve successive summers, successive in-service school years, or combinations of summer and in-service sessions. "Sequential-type institutes" are characterized by emphasis on coordinated series of courses in successive institutes, leading returning participants to a specific goal, such as a Master's degree. Award of grants to sequential institutes, however, does not imply downgrading of non-sequential institutes in the thinking of the Foundation. The Foundation position is that some institutes should provide coordinated sequences of courses for qualified participants, but other institutes should be available for participants whose needs and situations are best served by a "single-shot" institute. To avoid the appearance of preference for one type of institute over the other, a better name has been sought than the negative-sounding term, "non-sequential institute." "Unitary" seems the best that has been suggested thus far, so we now have "sequential institutes" and "unitary institutes." Unitary (or non-sequential) institutes are those which are designed primarily for a single summer (or in-service year) for a particular group of participants. A unitary institute might be offered only once; or it might be repeated several times, with little or great modification but primarily for different participants each time.

It is obvious that sequential institutes need assurance of support for several years, in order that sequences can be planned most effectively. But in many cases, planning for unitary institutes can also be more effective if it can extend beyond a single summer. The Foundation is experimenting with steps to provide assurance of support in successive summers for sequential institutes and for unitary institutes as well.

Although large sums of money are being provided through the National Science Foundation in support of institutes, praiseworthy and welcome support is coming also from other sources. The contributions of the host institutions and their faculties are great. Institutes and conferences are being supported by industrial concerns. (In some cases this support is being channeled into other academic areas. For example, General Electric now finances institutes for social science teachers instead of teachers of chemistry

and physics.) States and localities are becoming more active in providing subject-matter institutes for teachers in their jurisdictions.

It is earnestly hoped that still more of the resources needed to improve the teaching of science and mathematics will come from sources outside of the federal government. One of the summer institutes scheduled for 1961 for elementary school personnel stated in its local brochure that preference in that institute would be given to applicants whose school boards were willing to contribute $100 toward the operational costs of the institute. The institute specified that in each case the money must not be taken from the applicant teacher, but should be provided by the school board. It was hoped that a result would be that the school board would then have greater interest in seeing that the institute-acquired capabilities of its teacher would be used in appropriate assignment. Money from the NSF grant that was freed through the school board contributions to operating costs provided stipends for additional teachers to participate in the institute. Offers to contribute were received from more school boards than could be accommodated by the institute. But another type of response was also noted. This institute has been "reported" to the Foundation several times. Some citizens have an uneasy feeling that something is wrong when school districts are asked to share the responsibility for improving the competence of their teachers. They ask: "Isn't the National Science Foundation supposed to be paying for these institutes?"

The problem of improving the teaching of science and mathematics—and other subjects as well—is an enormous one. It is the responsibility of the entire nation, every citizen and group. It must not be left to the federal government alone.

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Review and future plans

Institutes in the Academic Year

The previous paper has reported on supplemental training activities for science and mathematics teachers encompassed in summer institutes and conferences. Beginning in 1956, this pattern of supplemental training was extended to programs which occur during the regular 'academic year. These comprise two major groups: 'academic year" institutes and "in-service" institutes.

This discussion is based on the NSF programs because they are representative of the most extensive activities at present, and specific data on these are readily available. This does not, of course, minimize in any way the credit which is due the numerous other activities supported by individual colleges, private funds, etc., ranging back to the institutes conducted by Huxley and Agassiz in 1871 or thereabout and including, for example, the General Electric and Shell Merit programs at present.[2] There are also other activities within the Foundation—such as certain fellowships, the visiting scientist programs, research participation, and conferences set up as special projects or "special field" institutes, and other government programs such as some of the NDEA fellowships and institutes of the U.S. Office of Education—which make significant contributions but which do not include large numbers of teachers.

Academic Year Institutes

The Academic Year Institutes were a natural outgrowth of the summer programs, and were instituted primarily to meet the increasing desire for training more adequate than just one summer. The academic year institutes are organized for groups—20–30 if in a single field, 40–50 if in several fields—of teachers who can take leave of absence and spend a year in full-time study. Often provision is made for work to continue during the summer session also, so that 30–36 semester hours of advanced work can usually be accomplished. The programs were initiated in 1956–57 with an institute at Oklahoma State under the direction of Dr. James Van Zant and one at Wisconsin under Dr. Harvey Sorum.

Table 1 shows some data on the growth of such institutes, and data on the types of participants are given in Table 2. It may be noted that secondary school teachers constitute about 95% of their participants at present.

Now that it is becoming possible for a teacher to carry out a longer program in summer also by study in a sequence of summers, it is important to recognize that the academic year programs complement the summer in significant qualitative aspects. In the academic year, a student has opportunity to accomplish the

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[2] Bacon, E. K., J. CHEM. EDUC., **38**, 39 (1961).

Table 1. Growth of Academic Year Institutes

Year	No. of proposals	No. of grants	No. of participants	Total amount of grants
1956–57	2	2	95	$ 504,700
1957–58	22	16	775	4,065,000
1958–59	33	19	925	4,906,500
1959–60	57	32	1508 HS 16 College	8,632,400
1960–61	65	33	1491 HS 43 College	9,210,600
1961–62	66	43	1494 HS 75 College*	9,794,300

* 1961–62 includes for the first time three institutes for college teachers only: one each in radiation biology and mathematics and one for junior college chemistry teachers at the University of the Pacific under direction of Dr. Emerson Cobb.

Table 2. Participants in Academic Year Institutes (1961–62 Estimations)

Distribution of participants by discipline (estimated)			Distribution of participants by school level (estimated)	
	Number	*Per cent*		
Biology	278	17.8	Junior high school teachers	179
Chemistry	248	15.9	Senior high school teachers	1303
General science	183	11.7	Pre-service secondary teachers	12
Earth science	34	2.2	College teachers	75
Mathematics	632	40.0		
Physics	194	12.4		
Total	1569		Total	1569

necessary readjustments to college life and study with ample time still remaining for much effective work then to be done. There are usually differences between the long-session and summer programs on individual campuses, which are reflected in somewhat different institute programs. There is also a difference between a program of steady study for nine months and one which is interrupted several times for intervals of nine or ten months, just as there is a difference between studying certain topics simultaneously and in sequence after such intervals.

The academic year institutes take a teacher out of the classroom for one year; but that teacher can return the next year with a master's level background—instead of four or five years later as would be required with summer work alone. Similarly, the academic year institutes permit observation of a new degree plan, etc., within one year rather than five. And there is often more contact and interchange with regular faculty—and thus more impact on programs of the institution itself—in the academic year. (One director recently noted that his 48 participants in 1959–60 worked with 58 different faculty members.)

Attempts to evaluate the success of the Academic Year Institutes involve all the usual complexities and uncertainties of such evaluations. It can be stated that both past participants and their colleagues and supervisors rate their experience as being beneficial. Participants are in demand on leaving and often receive premium salaries. There have been numerous instances of citation of such experience in job specifications, etc. And they do return to teaching (contrary to fears which some persons had expressed in the past) though a number may take college rather than high school places.

In-Service Institutes

The In-Service Institutes are usually organized in the familiar pattern of extension work, comprising special classes scheduled in evening hours, Saturday mornings, or late afternoon, so that persons currently teaching full time may participate. Occasionally a teacher may have "released time" from his job, but usually the institute is extra work and has to be limited to one course or about six semester hours of credit in a year. This program was started with two institutes in spring 1957: one in chemistry and four other fields at Antioch College under the direction of Dr. James F. Corwin, and one in chemistry and mathematics at Reed College under the direction of Mr. Gwen L. Taylor.

Summaries of data on the growth of in-service institutes are given in Tables 3 and 4. In 1959 a limited program for elementary teachers was begun. These do not usually emphasize chemistry as a separate field, but it is commonly a major component of work in physical science for these teachers.

The in-service institutes, in their turn, are different in certain qualitative aspects from other programs. Because of the time factors involved and the general working conditions, there is less concern about credit

Table 3. Growth of In-Service Institutes

Year	No. of proposals	No. of grants	No. of participants	Total Amount of Grants
1956–57 (Spring semester only)	3	2	90	$ 11,900
1957–58	60	21	635	162,300
1958–59	146	85	2,985	610,800
1959–60	225	182	8,690	1,941,700
(Elementary)	36	11	309	60,000
1960–61	250	191	8,888	2,173,900
(Elementary)	43	13	405	74,000
1961–62 (estd.)	304	236	10,816	2,701,800
(Elementary)	75	24	699	136,030

Table 4. Participants in In-Service Institutes (1961–62 Estimations)

Discipline	Number of participants	Per cent
Mathematics	6,236	57.7
Biology	1,398	13.0
Chemistry	795	7.3
Physics	1,114	10.3
Earth science	578	5.4
General science	230	2.1
Physical science	385	3.5
Oceanography	25	0.2
Radiation biology	55	0.5
Totals	10,816	100

hours and, often, quite frankly less concern about quantity of material covered. The work can, however, be correlated quite closely and effectively with teaching problems because of the close juxtaposition with actual classroom work. These institutes also reach, frequently, a teacher population quite different from those who can attend summer and academic year institutes.

Again the reports on effectiveness of the programs are encouraging. There need be little concern here about the sincerity of participants because they receive only

minimal allowances for travel, and the working conditions are hardly such as to attract the lazy!

Future Role of Institutes

Summer, academic year, in-service—each has made its contribution; together they have become and should continue to be effective methods for science and mathematics departments to contribute to the training of science and mathematics teachers. With due allowance for the problems of evaluation already noted, we have nonetheless uniformly encouraging evidence that the basic pattern of scientists and teachers working together is a successful one. But our major concern here should not be to recount past success so much as to consider future developments—to try to determine whether there is continuing need for these programs and, if so, how they may be improved.

The obvious starting point for determining current needs for institute-type training would be to know the qualifications of our present group of science and mathematics teachers; but only limited data are available on these. The U.S. Office of Education and the AAAS and NSF are trying to learn more; but in the meantime, it is necessary to adopt the engineering viewpoint of doing the best one can with present data, and with due caution that these estimates must be revised as new figures become available.

In Table 5 are summarized data (based on figures made available through the courtesy of the U.S. Office of Education based on a preliminary summary of studies for 1957 and 1958) on secondary school teachers and the source and extent of their training, with estimates of the amount of training they still need to reach the levels of professional background recommended by groups such as the Garrett subcommittee of the AAAS Cooperative Committee on Science and Mathematics Teaching and similar committees of other professional societies. These all follow a basic pattern which requires essentially a five-year program reaching the level of a master's degree for full professional status. Figures for the present NSF programs are given for comparison. (A "man-year" of study or an "academic year equivalent" (aye) is essentially one full academic year of work, about 30 semester hours. It takes about 4 summer or 5 in-service institutes to give one such equivalent of work.)

We have tried to project this need for basic training over a ten-year period, with allowances for population increases, etc., and have got a figure of some 30,000 aye per year. Even if we neglect the older group and try just to train the new teachers each year—the newly certified and those converted from other fields—some 20,000 aye per year would be needed.

Most important of all, we must continue to give most serious attention to the problem of keeping up to date in science, a problem which is continuous even for the best trained of teachers. It is in this area that the cooperation of active scientists and classroom teachers, which has been the key feature of institutes programs, is uniquely necessary. In time we can eliminate the need for remedial programs for basic training by improving undergraduate teacher training; but the "sabbatical-year" type of study needed to maintain a teacher's competence in science will represent a continuing need for supplemental work and training.

The experience of practice in many school systems plus the rate of growth of scientific knowledge (variously estimated as doubling every 8–12 years) indicates that one summer every four years is a fair estimate of the study needed to keep good teachers from losing touch, and thus to avoid drifting back into a situation we are just now beginning to alleviate. For 159,000 teachers this would require some 10,000 aye per year. In a 10-year period, 12,000 aye may be needed each year to allow for the increasing teacher population.

Thus 32,000 to 42,000 total aye per year are needed, depending upon whether we try to give basic training estimated on the needs for new teachers only or for all. These estimates are conservative[3]; despite the evident uncertainties in them, comparison with some 8500 aye now available shows that there is no question of a continuing need for institutes and other supplementary training for teachers. If any real *gain* is to be accomplished, it seems evident that we must have sharply increased efforts and that increased support should be sought from all possible sources. In spite of the major efforts which are now being made and the very real progress which has resulted, we are in danger of actually losing ground again, in these days of rapidly growing school and teacher populations, unless expanded and more effective programs can be developed.

If there is to be further growth and expansion in institutes programs, several points must be considered. First of all, it is evident from the figures above that we must emphasize whenever possible the importance of giving good basic training initially so as to eliminate the heavy remedial factor in present programs. The needs are so extensive that we cannot afford the waste

Table 5. Teacher Population and Training Needs
Secondary School Teachers (Grades 9–12)
Science and Mathematics

Current population by training		Estimated further training needed	
Lacking bachelor's	4,000	8,000 aye	
Bachelor's degree only	90,000	90,000	
Master's degree, without science major	52,000	26,000	
Master's degree, with science major	13,000		
	159,000	124,000	
By Experience		**Present Programs**	
Carried over from 1959	134,300	Academic yr. inst.	1494 aye
New teachers certified	8,200	Summer inst.	4429
"Converted" teachers	16,500	In-service inst.	1778
	159,000		7701
		NSF fellowships, NDEA fellowships, MAT, etc.	800 (estd.)
		Total	8500

[3] A report just issued by the National Education Association, "Teacher Supply and Demand in Public Schools," 1961, indicates that the annual rate of loss of teachers decreased somewhat last year and the supply of new teachers increased. However, even if this favorable trend continues for the next five years, the need noted above will be decreased less than 10%.

of poor professional training. Increased efforts and new ideas are needed for the further revision of teacher education programs over the country and, even more, for attracting good students into strong programs now available.

Furthermore, our efforts quite evidently cannot be restricted to any one agency alone. Support of state agencies or local school systems or industry are all needed as much as ever, in addition to that of the National Science Foundation and other federal agencies.

It is evident also that, now that we have had a reasonable period of experience which has demonstrated the basic general effectiveness of institutes, we need to re-examine carefully the contributions which each type can make so that the investment in the total program can attain maximum net efficiency in improving education in science and mathematics.

Particular attention should be given to developing more the shorter, lower cost programs, particularly in-service. There has been a strong tendency to gear programs to a 3-semester-hour course because of the common use of these for degree credits. But how important is this, for example, in the programs for "sabbaticals," i.e., those planned to keep teachers up to date? Here frequency of opportunity may be more critical than length and credit is not important. Could not shorter in-service programs be used to much advantage?

New patterns of approach are needed which may "multiply" the impact of institute training—for example, by work with supervisors or "key" teachers who may in turn assist other teachers in the local school systems.

The importance of increased attention to institutes for college teachers must be recognized. College faculties have been rightly concerned with the quality of high school programs, but now, with the flood tide of enrollments coming on, they must really work to keep their own houses in good order. Usually the needs of college teachers for advanced training have been met by individual programs supported by numerous fellowships; but in recent years many college teachers are starting out with bachelor's degrees only. For these, institutes leading to master's degrees are worth while because the work needed includes much group work in courses, and these are commonly not offered in summers. Furthermore, increased numbers of trained college teachers will be needed for the expansion of secondary school teacher programs noted above. (Last year 52% of the institutions giving degrees in science and mathematics filed proposals for institutes and 36% received grants.)

The burgeoning interest in science and mathematics for junior high school and the elementary schools is an indirect but nonetheless highly significant factor also. This will result in needs for supplementary training of teachers[4] which will require some of the available resources of participant support and help from college science faculties.

Individual institutes must be carefully re-examined to make sure that each may do the best job possible. In earlier years institutes could be planned on the simple assumption that all participants would need training in fundamentals of science or mathematics. This is still needed for many; but there are numerous other teachers who are now interested in progressing beyond beginning levels. Some institutes are needed for teachers specializing in a field and capable of advanced study therein, as well as for beginners; and it is critical that these be clearly differentiated in advance for the sake of potential applicants so as to avoid the waste (in time of the participants and of the faculty and in funds for their support) which is entailed when a teacher enrolls in an unsuitable program.

In this respect there is much to be done in the field of chemistry. So far there has been relatively little planning for teachers at various levels or for any sequence of work. Directors' reports from current institutes have noted this as one of the most common sources of difficulty. There are numerous programs in "Recent Advances" or "Modern Chemistry," but how many teachers may find these to be "deadends?" Where, for example, does the teacher turn after he has had an institute planned for general science teachers and finds he is interested in further study of chemistry? Careful planning, and some coordination among chemists over the country, are needed so that each institution may operate at levels where it can be most effective and so that at the same time reasonable distribution of effort may be maintained to meet the needs of teachers for advanced work or a sequence of courses as well as for study of fundamentals.[5]

In retrospect and prospect, therefore, it may be seen that the experiment of the institutes programs—one which has involved efforts of many people and many agencies and which has, most important of all, been based upon cooperation of scientists and teachers—has proved notably successful in past years. There is a very real need for continuing these efforts, but with some major problems to be faced.

These problems are clearly *critical* today: first, the continuous strengthening of the training of new teachers; second, wider and more effective use of low-cost programs such as in-service activities; third, new patterns which may reach more teachers; fourth, the development of really strong programs for college teachers; and fifth, eliminating undue overlapping and establishing clear-cut sequence and purposes in all the institutes programs; plus of course the continuing need for trying new ideas and new experimental programs such as those for elementary teachers. The combination of the situation still existing in our schools and colleges plus the waves of student population coming on makes the immediate future years a time when we must now strive to make even more extensive and effective use of the various patterns which have proved successful. Full cooperation and support from private sources, from local school systems, and from state and federal agencies will be needed.

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[4] See "Science Teaching in Elementary and Junior High Schools," report of a study by the American Association for the Advancement of Science, May, 1961.

[5] See, for example, "A Conference on Mathematics Curricula in Institutes," *American Math. Monthly*, **68**, 33 (1961), and "Recommendations for the Training of Teachers of Mathematics," report published by CUPM Committee, Mathematics Association of America, January, 1961.

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Contemporary Analysis and the Chemistry Curriculum

During recent years there has been much discussion about the curriculum in chemistry. Primarily as a result of extremely rapid accumulation of new chemical information, there has been pressure to include more and more of that new information in the curriculum. On the other hand, one can only go so far in adding new courses before the approach breaks down because of time limitations. Then, the only recourse is to make a critical evaluation of the material, not only within a given subject but also throughout the entire chemistry curriculum. Once the important facts and concepts have been decided upon, the next step is to assemble them into related groups so that they can be taught efficiently and effectively.

Currently there is much attention focused upon analysis. There seems to be agreement that it should be retained as part of the education of a chemist, but there is serious disagreement as to whether or not a separate subject is justified. The purposes of the present discussion are to suggest that: (a) there are a sufficient number of ideas and concepts involved in making quantitative measurements to be profitably presented as a separate unit; (b) disagreement about the need for a separate analytical course stems largely from the fact that most of the existing elementary analytical courses bear little relationship to modern analytical chemistry as it is practiced.

In broad terms, analytical chemistry is the application of chemical principles and facts, aided by tools of physics and electrical engineering, to problems of measurement concerned with chemical composition and structure. If one accepts such a definition, it is clear that analysis must be taught rather late in the curriculum if it is to consist of much more than practice in manipulations and presentation of a few greatly simplified and generalized concepts. For example, there is nothing particularly difficult about the concepts involved in quantitatively precipitating a pure compound or of applying the Lambert-Beer law to an energy-absorbing system—and obtaining an indefinite number or significant figures in the answer. However, quantitative analysis, by illustrating actual problems and their solutions, can demonstrate conclusively to the student that real systems require more than just deft manipulation or extra care in order to convert a qualitative observation to a quantitative one (*1*).

Another goal of analysis, which ranks second only to getting a result of acceptable accuracy and precision, is to obtain the result with the expenditure of a minimum amount of effort and elapsed time. It is insufficient to know that a particular separation, identification, or determination can be made if the chemist is persistent. In brief, efficiency ranks close behind reliability in determing the value of an analytical method.

In the discussion that follows, emphasis has been placed upon quantitative analysis. However, in attempting to define the role played by quantitative analysis in the modern curriculum, it is helpful to examine the earlier roles played by both qualitative and quantitative analysis.

What Has Happened to "Qual"?

The classical undergraduate subject of qualitative inorganic analysis was once very comprehensive in scope and included analyses of complex natural and industrial products. During the course, the student became acquainted with a large fraction of the known reactions of chemical species in aqueous solutions. In view of the fact that as a practising chemist he would later have to do most of his own identifications, the course equipped the student with most of the necessary chemical facts and techniques for performing his job. Much the same could be said for his training in quantitative inorganic analysis and in organic elemental analysis. Unfortunately, the terms *qualitative* and *quantitative* analysis have been retained over the years, while the contents of the courses have been severely cut. Meanwhile, the number of available (but unused) reactions and tools has greatly increased. To the writer, the use of the term "analysis" implies that the reactions are *widely* or *routinely* used for making analyses rather than the fact that they *might* be or are only *occasionally* used. Instead, courses in qualitative analysis, particularly those in inorganic, bear little resemblance to practice. In part, this is due to a change in goals. Today the schemes are used to illustrate the systematization of reactions according to periodic relationships and to teach equilibrium concepts. Although the present abbreviated courses appear better adapted to those ends than to analysis, there is reason to believe that equilibrium can be explored more completely in quantitative experiments and that the fragmentary remains of the classical qualitative scheme do not satisfactorily illustrate chemical periodicity.

The academic emphasis on chemical reactions to the exclusion of instruments, represents another discrepancy in relation to actual qualitative analysis. If instruments are included in a course presented in the first or second year, as is the case in some organic qualitative courses, the student is exposed to a more realistic situation. Realism, however, has to be balanced against his lack of complete understanding of the true meanings (and limitations) of the data from the unfamiliar "black boxes."

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The final discrepancy between academic and actual qualitative problems results from the fact that the analyst is rarely ever faced with a completely unknown sample requiring an identification, such as that of a new biologically active substance from a natural source. He is much more likely to be faced with the need for determining the presence or absence of a particular substance or functional group in a mixture of known or suspected substances. Hence, it seems inevitable that academic courses in qualitative analysis be considered as devices for systematizing reactions rather than as practical schemes. To avoid misleading the student, the term "analysis" should be dropped or the entire name changed.

Did "Instrumental" Modernize "Quant"?

Much the same can be said about the content of the classical course in quantitative analysis. However, the addition of courses in "instrumental analysis" has prevented the over-all treatment of quantitative measurements from becoming completely outmoded. It should be recognized, nevertheless, that instrumental courses can be quite unrepresentative. One factor is the instructor's inability to justify the time required for a student to gain first-hand familiarity with all of the important existing instruments. A second factor is the high cost of some of the most useful instruments, such as nuclear magnetic resonance, X-ray and mass spectrometers. The cost of each severely limits the probability of finding a single instrument of a given type in a small college or of finding a sufficient number in a large university to permit individual use by students in a class. Hence, an instrumental course may emphasize more readily available apparatus.

The approach we have used in instrumental analysis to counteract such a deficiency is to group together several related instrumental methods and to restrict discussion to the fundamental problems common to each. For example, the problems of making quantitative measurements of energy absorption are largely independent of the region of energy considered; sample preparation, effects of the degree of monochromaticity of the energy, and differential techniques for improving precision are shared in common by X-ray, infrared, and other regions of energy, particularly with respect to a single absolute measurement on a sample. Similarly, titrimetric methods involving absorption share a large number of problems with one another and with continuous measurements on process streams. We have adopted a similar approach for measurements involving emission and scattering of radiation. Treatment of the electrical properties of chemical systems is susceptible to analogous treatment (*2*).

With regard to the over-all subject of quantitative analysis, the writer believes that a combination of chemical and instrumental principles, with spare use of typical examples, can profitably consume the amount of time now devoted to the total course effort on chemical and instrumental methods. On the other hand, a formal division of subject matter along those lines seems undesirable. One reason is pragmatic: the course in quantitative analysis is a terminal requirement for most scientists and engineers who are non-chemists, a group which is relying increasingly upon the use of instruments in later work. In order to counteract the deficiency, some colleges have long incorporated in the elementary course two or three simple experiments on subjects such as potentiometry and colorimetry. Unfortunately, if the course is scheduled in the second year of the chemistry curriculum, the treatment of instrumental problems as an integral part of analysis must necessarily be superficial, both in lecture and laboratory.

A second reason for combining chemical and instrumental approaches is based upon the belief that, provided the student has already had some contact with instruments in physics or physical chemistry, a more fundamental unified approach to the over-all problem of quantitation in non-ideal chemical systems is possible. This will be discussed below in more detail under representative subjects such as sampling and standardization. In essence, such an approach would restore current academic practice to a place corresponding to that filled by the classical course many years ago.

Emphasize Sampling

Sampling is a topic of vital importance which, because it is far from glamorous, is usually given very brief attention at the end of a quantitative course—if it is mentioned at all. The writer suspects that a substantial fraction of samples submitted for quantitative analysis give misleading or limited information because the chemist lacked appreciation for this factor in taking his original sample (*3*). Instead of treating the topic of sampling as a poor relative, the teacher of analytical chemistry must face his responsibility.

To add variety as well as perspective to the subject, new problems can profitably be incorporated such as contamination and solute loss. For example, solute loss by adsorption onto the walls of the storage bottle or pipet is a difficulty encountered frequently in studies using very dilute solutions of radio tracers (*4*). Similarly, in the plant equipped for automatic "on-line" analyses for water in process streams, a small or momentary increase in concentration may be largely obscured by losses to the wall of the pipe connecting the stream and the analyzer; in turn, desorption may obscure a decrease.

The statistics of sampling is another aspect that deserves emphasis. In view of the trend toward micro analysis, particularly with respect to the small amounts of material regularly submitted for carbon and hydrogen determinations, an appreciation of sampling statistics is not only valuable but imperative.

Standardization Is Fundamental

Another important concept that could be discussed more realistically by combining chemical and instrumental methods is that of standardization, including the related matter of "blanks." The modern analyst, in addition to the classical aspects related to the calibration of chemical stoichiometry, is faced with instruments which introduce additional similar demands. In most courses in instrumental analysis, discussions of standardization tend to be restricted to

the instrument of the moment, rather than dealing with the problem in a general fundamental fashion so that the principle can be applied with little change to diverse instruments such as a polarograph and a flame photometer. Provided the instrument response is moderately stable, the method of "standard addition" may be employed. If sodium is to be determined in blood using a flame photometer, the sample is analyzed both before and after a known amount of sodium has been added. This not only permits calibration of the response directly in the matrix of the sample but may also shorten the total time required for the successive steps of calibration and determination. If variation in instrument response or in physical condition of the sample occurs within times that are shorter than that required for a measurement, the "internal standard" method can be applied. In the case of the blood sample, a standard amount of lithium is added because it behaves much like sodium, it is known not to interfere, and it is ordinarily not a constituent of blood. Both sodium and lithium are then determined using the latter as a standard. Both methods are often applicable in correcting for non-ideal behavior, but sometimes one must choose between the longer time required for the standard addition procedure and the somewhat greater complexity of the instrument usually required for an internal standard measurement. To complete the discussion of calibration, one can describe the particularly difficult problems faced by the analyst in attempting to make automatic on-line analyses.

Broaden the Discussion of Separation

A third important factor that can be gained from elementary quantitative analysis is an appreciation for the chemical and physical factors involved in the quantitative isolation of a pure substance. The classical course deals with this matter at length but the subject matter is often restricted almost entirely to precipitation as a form of separation. Admittedly, there are many interesting features involved in precipitation and co-precipitation, but the student of today, who is not exposed in equal detail to techniques such as ion exchange, liquid-liquid extraction, and gas chromatography, is certainly being grossly misled. In giving a broader treatment, it is helpful to remind the student that a single thermodynamic principle governs the equilibrium distribution of a solute between any two phases. Although the teacher cannot dwell upon the descriptive features of individual methods in any detail, he should certainly stress the fundamental aspects common to all chromatographic procedures. For example, theory is now available which permits the chemist to calculate an optimum ratio of the volume of the two phases for a particular separation (*5*). Furthermore, his selection of a procedure, if based upon earlier study of the peculiar advantages and disadvantages of each type of general separation procedure, should minimize the time lost by the chemist who, for example, struggles for days or weeks to develop a new gas-chromatographic separation for a system that can be handled successfully in a few minutes by a different approach, such as liquid-liquid extraction or ion exchange. In conclusion, it is important to note that, although the descriptive information about separations can be taught at any time, separation theory which is based upon simple probability concepts can be most efficiently taught after the class has been exposed to the statistical tests mentioned below.

What Does Only One Result Mean?

During the past two years, the writer has had the opportunity to test his idea that the use of simple statistical procedures should be given a place in elementary quantitative analysis. In view of the fact that the chief, if not the sole, product of an analysis is a number, it is vitally important that the analyst be acquainted with statistical procedures for comparing the accuracy and precision of his methods and also for evaluating the data obtained for a particular sample. The t, F, and Q tests permit the analyst to make such decisions. The Q test deals with the rejection of a suspected result when the number of replicates is small (*6*). Judgment is made in terms of the range rather than the average (or standard) deviation. The t and F tests are used to determine the probability that two sets of results (obtained using different procedures) are members of the same population (*7*). The t test is used for comparing the two means, while the F test is used for the variances (spreads). When the calculated probability is low, the results from the two procedures are "significantly different." Furthermore, if the analyst is compelled to devise and judge a new procedure, an evaluation of variables can be performed most efficiently if he has been introduced previously to the statistical design of experiments.

It may be helpful at this time to illustrate how a limited knowledge of statistics can be combined with the use of liquid unknowns to give the student better insight with respect to the meaning of quantitative results. In the past, liquid unknowns have been used by some instructors because they were easy to prepare and convenient to dispense. The use of liquids has also permitted a wider variety of analyses to be done in a shorter time than if difficultly soluble substances had been used. Whether the instructor uses solid or liquid unknowns, the student rarely has any feeling about how well he has done except as it is reflected in a numerical grade and a vague statement to the effect that "a good result for this unknown should be accurate and precise within 0.3% relative." If a limited number of solid samples is available to the instructor, it is unwise for him to divulge the correct answer. On the other hand, when an infinite number of different volumes (measured out by means of a buret) from a single solution can be handed out as unknowns to the students, each student can be told at once the answer for his determination and, in addition, can later see the assembled results from the entire class. Although we have not as yet broken with tradition to the point of immediately telling the student how his result compared with the accepted result, a class has been assigned the exercise of calculating the mean and the standard deviation for the gravimetric determination of sulfate, using the normalized values submitted by each individual in the class. Student response was excellent.

The staff, at the suggestion of S. G. Simpson, has long handed out special unknowns of non-routine nature for the last few periods of the laboratory. Last year,

a variation of the above approach was tested for the writer by three students who chose to analyze non-routine unknowns in two different ways in order to compare the precision of the two procedures and the agreement between the mean values. Similarly, one student compared the normalities for a given solution of sodium hydroxide that had been standardized (using weight burets) against two different primary standards. In the process of learning that measurable differences might exist between accepted standards, he also gained new insight with respect to the meaning and interpretation of results. Another variation that suggests itself is to have a student analyze a non-routine unknown and evaluate the effect of one or two experimental variables at two different levels of the effect. Though this work may be done at the cost of one or two conventional experiments, the student should be much better prepared to interpret his own experimental results at a later time when no one is available to provide a "known" answer on which to base the acceptability of his results.

Organic Analysis As an Integral Part of "Quant"

A final remark about the general content of the course seems in order. There is growing recognition of the fact that organic chemistry occupies the efforts of approximately half of the practising chemists. In order to give the student a representative picture of modern analysis, as well as to draw upon material that should appeal to the interests of a large fraction of them, quantitative reactions of covalent organic compounds (which often require approaches different from ionic compounds) should be given a prominent place (*8*). Most inorganic redox reactions are ionic and rapidly go to completion, whereas organic ones (bromine, periodate) are not (*9*). Furthermore, if an organic compound is allowed to remain in contact with excess reagent for an extended time, side reactions often become significant. Unfortunately, while the usual first course in chemistry introduces the student to a moderate number of elements, the discussion of organic chemistry is often quite brief. When the writer attempted to introduce, at Stanford, quantitative functional group determinations of organic compounds to second-year students who had no background in organic chemistry, the methods had to be treated in cook-book fashion. However, experience at MIT and in other schools has shown that when organic chemistry precedes the course in quantitative analysis, the mystery and the routine disappear.

Keep the Analysis Course

Before concluding the discussion, the writer would like to consider two reasons that have been given for eliminating separate analytical courses from the formal training of a chemist. The first reason is concerned with the fact that, because analytical chemistry is admittedly the application of physical chemistry to problems of composition and structure, it should be omitted or postponed from the curriculum in order to make room for "fundamental information." In contrast, the writer believes that the principles on which quantitative measurements and efficient separations of chemical species are based represent a coherent set of fundamental ideas that can be treated most effectively in a separate course. The course in quantitative analysis not only emphasizes the omnipresent nature of chemical and instrumental deviations resulting from non-ideal behavior but also provides the student with typical basic approaches for minimizing their effects. When provided with a limited knowledge of statistics, the student can evaluate all types of errors, including those arising from deliberate changes of suspected variables, with greater certainty than otherwise. At the same time, a separate course in quantitative analysis during the junior or senior year can provide an excellent opportunity for synthesizing the students' previous experience in organic, inorganic, and physical chemistry. The course also helps the student to realize that when the sampling has been carefully performed and the analytical procedures are sufficiently precise, quantitative information (such as an elemental analysis for carbon) can provide useful qualitative information for identification of a compound.

A second reason that has been given for eliminating a separate "quant" course is that the necessary material can be integrated with other courses. In order to answer that point, it seems well to return to an earlier statement concerning the effective and efficient division of material. There is no question that the ultimate goal of training a chemist, particularly at the undergraduate level, is to produce a well-rounded student without qualification as to whether he is organic, inorganic, biochemical, physical, or analytical. Likewise, it is generally recognized that some concepts can and should be grouped together for efficient presentation at the risk of a momentary loss of the overall picture. To the writer, it appears that the synthetic organic or inorganic chemist is rightly more concerned with the preparation either of a new substance or of an known substance by a new procedure. It seems inappropriate to force a semi-quantitative, much less a quantitative, approach upon courses in synthesis because it would severely limit the scope of the material that could be covered. It would also becloud the main issues at hand: the philosophy, reactions, mechanisms, and techniques of producing a new substance. Much the same would appear to be true for courses of physical chemistry where the emphasis is upon unifying as many facts as possible by utilizing broad principles which assume ideal or nearly ideal behavior. The time required to discuss details relating to quantitative measurement or the behavior of non-ideal situations would be diverting and would tend to obscure the main goal. The fact that many important chemical conclusions can be reached without relying upon precise quantitative measurements is one reason why some chemists do not recognize the vital role played on other occasions by quantitative analyses. In addition the large volume of routine analysis that is performed tends to obscure the need for ingenuity and imagination in solving the non-routine, complex, compositional problems that demand chemical research of high caliber (*9*). As a matter of fact, there are many chemists of other classifications who are actually contributing little new in the way of novel ideas. A bit of reflection will generally lead to the conclusion that syntheses of new compounds or reporting of new physical data are invaluable, but that

time-worn methods (or slight modifications) are being employed for the most part. If the over-all problem of making, identifying, and observing the behavior of a new compound is considered, a chemist should recognize that all three aspects are mutually important, are mutually interdependent, and can be equally challenging.

The above arguments, although negative in one sense, appear to support the need for a separate course in analysis. In addition, the writer submits a positive reason: the realization that the necessary fundamental ideas can be grouped efficiently for discussion.

If the need for a separate course is accepted, one vital aspect remains to be recognized. Because there is general agreement that *any* course will be taught most effectively by those who are actively interested in it and strongly believe that the subject is important, it is clear that such a course should be taught by an analytical chemist.

Acknowledgment

In addition to MIT colleagues and former students who have patiently endured long discussions that have brought these ideas into focus, the writer wishes to thank C. E. Bricker, M. G. Burford, N. H. Furman, John Mitchell, Jr., and R. F. Stalzer.

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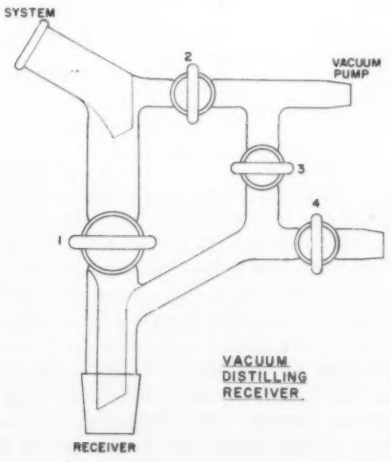
Fractionation Technique for Vacuum Distillation

The receiver shown in the figure has been found to be of great advantage in operating a fractional distillation under vacuum during which repeated small fractions had to be collected. During the course of a distillation the stopcocks numbered in the figure were in the open (*o*) or closed (*c*) condition: 1 *o* or *c*, 2 *o*, 3 *o*, and 4 *c*. When the operator wishes to remove a fraction, first stopcock 2 is briefly (2 sec) closed to suck down the small slug of liquid adhering to the glass above stopcock 1, and then the stopcocks are adjusted as follows: 1 *c*, 2 *o*, 3 *c*, and 4 *o*. The distilling receiver containing the fraction is removed and replaced with an empty distilling receiver. An auxiliary vacuum pump is connected to the open tube on the right of stopcock 4. The receiver is evacuated by the auxiliary pump until the characteristic sound of the pump working against an evacuated system is obtained. Then the stopcocks are changed in the following order: 4 *c*, 3 *o*, 1 *o*. The auxiliary pump is removed and the distillation continued. The operation could be simplified by using a three-way stopcock for stopcock 4, which could open the receiver either to the atmosphere or to the auxiliary pump.

This technique has been used satisfactorily for the vacuum distillation of a variety of high-boiling organic compounds with various columns. Decomposition in the distilling pot has been minimized and pressure readings have remained constant for several fraction changes.



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Calculating Molar Solubilities from Equilibrium Constants

As a part of the study of qualitative analysis, students in the first or second year of chemistry are introduced to the calculation of solubilities from solubility products. The usual sequence is to calculate molar solubilities of salts in pure water from solubility product constants and vice versa, then to calculate the effect of introducing a common ion, and eventually, after polyprotic acids and hydrolysis have been studied, to worry about the effects of hydrolysis of the anions. Cation hydrolysis is usually not treated quantitatively at all.

The recent compilation of equilibrium constant data for ionic solutions[1] makes data available for many interesting calculations which are not too difficult for the best students of elementary chemistry to tackle. The problem of calculating molar solubilities can sometimes become extremely complicated if all the factors are taken into account. Fortunately, there are a number of fairly insoluble salts which are relatively free from these complications, and from the point of view of making the calculations realistic, it is preferable to deal with these salts as examples for the students.

The simple approach to the problem, as given in most texts, is as follows: A salt M_aX_b dissolves according to the reaction

$$M_aX_b(s) \rightleftharpoons aM^{+b} + bX^{-a}$$

and the equilibrium constant governing this reaction is the solubility product constant (square brackets indicate concentrations in mole per liter).

$$K_{sp} = [M^{+b}]^a\,[X^{-a}]^b$$

The conventional approximation to make is that only M^{+b} and X^{-a} exist in the solution, and that if the salt is dissolved in pure water, there is no other source of the ions. Then if S is the solubility in moles per liter (gram formula weights per liter),

$$[M^{+b}] = aS$$

$$[X^{-a}] = bS$$

and

$$K_{sp} = (aS)^a(bS)^b$$

Very often the discussion is carried no farther, and after drilling on several dozen problems like this the student is left with the feeling that this is all there is to it. Nothing could be further from the truth.

There are three classes of reactions which occur when a salt dissolves in pure water which may invalidate the conventional approximation used above: (1) Hydrolysis of the anion. Only salts of strong acids will be free from this difficulty, and most of these are too soluble. (2) Formation of complexes, particularly neutral molecules of the salt or ion pairs, but often more complicated ions. (3) Hydrolysis of the cation, which is the same as the formation of hydroxide complexes.

It is, in fact, fairly difficult to find salts for which the conventional approximation yields even the right order of magnitude for the molar solubility in pure water. Sparingly soluble substances which are salts of strong acids are rare, but a few are listed in Table 1 with their solubility products[1] and molar solubilities.

Table 1. Molar Solubilities and Solubility Products of Some Selected Compounds[1]

Compound	pK_{sp}[a]	Solubility (moles/liter)[e]
$SrSO_4$	6.21	7.9×10^{-4}
$BaSO_4$	10.01	1.0×10^{-5}
$PbSO_4$	7.80	1.25×10^{-4}
Hg_2SO_4[b]	6.32	6.9×10^{-4}
Ag_2SO_4	4.80	1.6×10^{-2}
MgF_2	8.19	1.15×10^{-3}
CaF_2	10.40	2.15×10^{-4}
SrF_2	8.60	8.5×10^{-4}
BaF_2	5.98	6.4×10^{-3}
CuCl	6.73	4.3×10^{-4}
Hg_2Cl_2[c]	16.88	1.5×10^{-6}
AgCl	9.75	1.35×10^{-5}
TlCl	3.72	1.4×10^{-2}
AgBr	12.34	6.8×10^{-7}
TlBr	4.81	3.9×10^{-3}
$Ca(IO_3)_2$	6.15	2.6×10^{-2}
$Ba(IO_3)_2$	8.82	7.3×10^{-4}
$La(IO_3)_3$	11.21	6.9×10^{-4}
$Ce(IO_3)_3$	9.50	1.85×10^{-3}
$Cu(IO_3)_2$	7.13	2.6×10^{-3}
$AgIO_3$	7.52	1.7×10^{-4}
$Hg_2(IO_3)_2$[d]	13.71	1.7×10^{-5}
$Pb(IO_3)_2$	12.49	4.3×10^{-5}
$TlIO_3$	5.51	1.75×10^{-3}
$AgBrO_3$	4.28	7.2×10^{-3}
$Mg(OH)_2$	10.93	1.43×10^{-4}
AgI	16.08	9.1×10^{-9}
TlI	7.19	2.5×10^{-4}

[a] $pK_{sp} = -\log_{10}K_{sp}$.
[b] $Hg_2SO_4(s) \rightleftharpoons Hg_2^{++} + SO_4^{--}$.
[c] $Hg_2Cl_2(s) \rightleftharpoons Hg_2^{++} + 2Cl^-$.
[d] $Hg_2(IO_3)_2(s) \rightleftharpoons Hg_2^{++} + 2IO_3^-$.
[e] Assuming activity coefficients are unity.

The sulfates of Sr, Ba, Pb, Hg(I), and Ag; the fluorides of Group IIa; the chlorides of Cu(I), Hg(I), Ag, and Tl(I); the bromides and iodides of Ag and Tl(I); the iodates of Ca, Ba, La(III), Ce(III), Cu(II), Ag, Hg(I), Pb, Tl(I); $AgBrO_3$ and $Mg(OH)_2$ all satisfy the restrictions of small solubility, small hydrolysis of anions and cations, and small amount of complex formation. Other compounds may possibly be found by careful study of the tables of stability constants.

[1] Bjerrum, J., Schwarzenbach, G., and Sillén, L. G., "Stability Constants," Special Publication No. 7, The Chemical Society, London, 1958.

To what extent is the conventional approximation satisfied even with these salts? The best-behaved of all is $BaSO_4$, as illustrated by the following calculation:

Example 1: $BaSO_4$

Find the concentrations of all species present in a saturated solution of $BaSO_4$. The reactions that take place and their equilibrium constants are:[2]

$$BaSO_4(s) \rightleftharpoons Ba^{++} + SO_4^{--} \qquad K_1 = 1.0 \times 10^{-10} \quad (1)$$

$$Ba^{++} + OH^- \rightleftharpoons BaOH^+ \qquad K_2 = 4.8 \quad (2)$$

$$SO_4^{--} + H^+ \rightleftharpoons HSO_4^- \qquad K_3 = 98 \quad (3)$$

$$H_2O \rightleftharpoons H^+ + OH^- \qquad K_w = 1.0 \times 10^{-14}$$

Including the molar solubility S, there are seven variables, so we require three more relations, provided by a mass balance on the barium in solution, a mass balance on the sulfate in solution, and the electroneutrality condition.[3]

$$S = [Ba^{++}] + [BaOH^+] \quad (4)$$

$$S = [SO_4^{--}] + [HSO_4^-] \quad (5)$$

$$2[Ba^{++}] + [BaOH^+] + [H^+] = 2[SO_4^{--}] + [HSO_4^-] + [OH^-] \quad (6)$$

If the extent of hydrolysis is small, $S = [Ba^{++}] = [SO_4^{--}]$, and from reaction (1)

$$[Ba^{++}][SO_4^{--}] = S^2 = 1.0 \times 10^{-10}$$

$$S = 1.0 \times 10^{-5}$$

To find the concentration of the other species, eliminate $[Ba^{++}]$ and $[SO_4^{--}]$ from (6) by using (4) and (5); this gives

$$[H^+] + [HSO_4^-] = [OH^-] + [BaOH^+] \quad (7)$$

From equilibrium 2, using $[Ba^{++}] = 1.0 \times 10^{-5}$,

$$\frac{[BaOH^+]}{[OH^-]} = (4.8)\ (1.0 \times 10^{-5}) = 4.8 \times 10^{-5} \quad (8)$$

which shows that $[BaOH^+]$ is negligible in (7). From equilibrium (3),

$$\frac{[HSO_4^-]}{[H^+]} = (98)(1.0 \times 10^{-5}) = 9.8 \times 10^{-4} \quad (9)$$

which shows that $[HSO_4^-]$ is negligible in (7); thus $[H^+] = [OH^-]$ from (7) and from the water ionization equilibrium $[H^+] = [OH^-] = 1.0 \times 10^{-7}$. Finally, from (8) and (9)

$$[BaOH^+] = (4.8 \times 10^{-5})\ (1.0 \times 10^{-7}) = 4.8 \times 10^{-12}$$

$$[HSO_4^-] = (9.8 \times 10^{-4})\ (1.0 \times 10^{-7}) = 9.8 \times 10^{-11}$$

Looking back at (4) and (5) we see that neglect of hydrolysis has introduced an error of only about 1 in 10^5 in S, which is very good. Neglect of activity coefficient corrections for ionic interaction and inherent errors in the data restrict the accuracy of this calculation to about 15%. For barium sulfate, then, the conventional approximation is very good.

Example 2: CaF_2

Find the concentrations of all species in saturated CaF_2. Hydrolysis of the cation is still very small, but since HF is a weaker acid than HSO_4^-, anion hydrolysis is greater than in Example 1. In addition, a complex ion is formed:

$$CaF_2(s) \rightleftharpoons Ca^{++} + 2F^- \qquad K_1 = 4.0 \times 10^{-11} \quad (1)$$

$$Ca^{++} + OH^- \rightleftharpoons CaOH^+ \qquad K_2 = 20 \quad (2)$$

$$F^- + H^+ \rightleftharpoons HF \qquad K_3 = 1.5 \times 10^3 \quad (3)$$

$$Ca^{++} + F^- \rightleftharpoons CaF^+ \qquad K_4 = 10 \quad (4)$$

A calculation similar to Example 1 yields the following results:

$$S = [Ca^{++}] = 2.15 \times 10^{-4} \qquad [H^+] = 7.8 \times 10^{-8}$$

$$[F^-] = 4.3 \times 10^{-4} \qquad [HF] = 5.0 \times 10^{-8}$$

$$[CaF^+] = 9.2 \times 10^{-7} \qquad [CaOH^+] = 5.6 \times 10^{-10}$$

$$[OH^-] = 1.3 \times 10^{-7}$$

and again we see that the conventional approximation is satisfied to better than 1%.

Example 3: AgCl

Find the concentrations of all species in saturated AgCl. This is perhaps the classic example for solubility calculations. Even though several complex ions are formed, their concentrations are small in pure water:

$$AgCl(s) \rightleftharpoons Ag^+ + Cl^- \qquad K_1 = 1.8 \times 10^{-10} \quad (1)$$

$$Ag^+ + OH^- \rightleftharpoons AgOH \qquad K_2 = 2.0 \times 10^3 \quad (2)$$

$$Ag^+ + Cl^- \rightleftharpoons AgCl \qquad K_3 = 1.1 \times 10^3 \quad (3)$$

$$AgCl + Cl^- \rightleftharpoons AgCl_2^- \qquad K_4 = 1.0 \times 10^2 \quad (4)$$

$$AgCl + Ag^+ \rightleftharpoons Ag_2Cl^+ \qquad K_5 = 1.5 \times 10^2 \quad (5)$$

$$AgCl_2^- + Cl^- \rightleftharpoons AgCl_3^{--} \qquad K_6 = 1.0 \quad (6)$$

$$Ag_2Cl^+ + Ag^+ \rightleftharpoons Ag_3Cl^{++} \qquad K_7 = 1.8 \quad (7)$$

$$H_2O \rightleftharpoons H^+ + OH^- \qquad K_w = 1.0 \times 10^{-14}$$

A mass balance on silver, a mass balance on chlorine, and a mass balance on OH^- (more convenient than electroneutrality here) complete the equations:

$$S = [Ag^+] + [AgOH] + [AgCl] + [AgCl_2^-] + [AgCl_3^{--}] + 2[Ag_2Cl^+] + 3[Ag_3Cl^{++}] \quad (8)$$

$$S = [Cl^-] + [AgCl] + 2[AgCl_2^-] + 3[AgCl_3^{--}] + [Ag_2Cl^+] + [Ag_3Cl^{++}] \quad (9)$$

$$[H^+] = [AgOH] + [OH^-] \quad (10)$$

If Ag^+ and Cl^- are predominant, from reaction (1),

$$[Ag^+][Cl^-] = S^2 = 1.8 \times 10^{-10}$$

$$S = 1.35 \times 10^{-5} \quad (11)$$

Combining reactions (1) and (3) gives

$$[AgCl] = [Ag^+]\ [Cl^-]\ (1.1 \times 10^3) = (1.8 \times 10^{-10})\ (1.1 \times 10^3)$$

$$[AgCl] = 2.0 \times 10^{-7} \quad (12)$$

Equation (2), with $[Ag^+] = 1.35 \times 10^{-5}$ gives

$$\frac{[AgOH]}{[OH^-]} = (1.35 \times 10^{-5})\ (2.0 \times 10^3) = 2.7 \times 10^{-3} \quad (13)$$

Thus [AgOH] is negligible in (10), and from K_w: $[H^+] = [OH^-] = 1.0 \times 10^{-7}$. Substituting this in (13) gives

$$[AgOH] = (2.7 \times 10^{-3})\ (1.0 \times 10^{-7}) = 2.7 \times 10^{-10}$$

The rest come easily: From reaction (4)

$$[AgCl_2^-] = (1.0 \times 10^2)\ [AgCl][Cl^-] = (1.0 \times 10^2)(2.0 \times 10^{-7})(1.35 \times 10^{-5})$$

$$[AgCl_2^-] = 2.7 \times 10^{-10}$$

From reaction (5)

$$[Ag_2Cl^+] = (1.5 \times 10^2)[AgCl][Ag^+] = 4.0 \times 10^{-10}$$

From reactions (6) and (7)

$$[AgCl_3^{--}] = 3.6 \times 10^{-15};\ [Ag_3Cl^{++}] = 9.7 \times 10^{-15}$$

Checking back in equations (8) and (9), we see that the conventional approximation is good to 1%.

In case one might be tempted to think that these examples justify the use of the conventional approximation in all cases, it is appropiate to cite here a number of cases where the conventional approximation is

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[2] As is conventional, solvent ligands are not written in the ionic formulae, i.e., H^+, not H_3O^+.

[3] BUTLER, J. N., J. CHEM. EDUC., **38**, 141 (1961).

often used, quite incorrectly. Outstanding examples are the metal sulfides usually encountered in qualitative analysis. Although the cases of practical importance for analysis do not involve the solution of sulfides in pure water, but rather in water saturated with H_2S at various degrees of acidity, one often finds textbook problems involving the solubility of sulfides in pure water solved using the conventional approximation. How wrong this can be is shown in the following examples.

Example 4: HgS

Calculate the solubility of HgS in pure water and the concentrations of all species. The equilibria involved are

$$HgS(s) \rightleftharpoons Hg^{++} + S^{--} \qquad K_1 = 1.0 \times 10^{-51} \quad (1)$$

$$Hg^{++} + H_2O \rightleftharpoons HgOH^+ + H^+ \qquad K_2 = 3.2 \times 10^{-3} \quad (2)$$

$$HgOH^+ + H_2O \rightleftharpoons Hg(OH)_2 + H^+ \qquad K_3 = 1.4 \times 10^{-3} \quad (3)$$

$$S^{--} + H_2O \rightleftharpoons HS^- + OH^- \qquad K_4 = 8.0 \times 10^{-2} \quad (4)$$

$$HS^- + H_2O \rightleftharpoons H_2S + OH^- \qquad K_5 = 1.0 \times 10^{-7} \quad (5)$$

$$HgS(s) + H_2S \rightleftharpoons Hg(SH)_2 \qquad K_6 = 6.3 \times 10^{-7} \quad (6)$$

$$Hg(SH)_2 \rightleftharpoons HgS_2^{--} + 2H^+ \qquad K_7 = 1.0 \times 10^{-7} \quad (7)$$

$$H_2O \rightleftharpoons H^+ + OH^- \qquad K_w = 1.0 \times 10^{-14}$$

The two mass balance and electroneutrality conditions are

$$S = [Hg^{++}] + [HgOH^+] + [Hg(OH)_2] + [Hg(SH)_2] + [HgS_2^{--}] \quad (8)$$

$$S = [S^{--}] + [HS^-] + [H_2S] + 2[Hg(SH)_2] + 2[HgS_2^{--}] \quad (9)$$

$$[H^+] + [HgOH^+] + 2[Hg^{++}] = [OH^-] + 2[S^{--}] + [Hg^-] + 2[HgS_2^{--}] \quad (10)$$

A few trial calculations quickly convince one that the main species in solution are H^+ and OH^-, and next after those in importance are HgS_2^{--} and $Hg(OH)_2$. This means that we have from (8), (9), and (10), using K_w,

$$S = [Hg(OH)_2] + [HgS_2^{--}] \quad (11)$$

$$S = 2[HgS_2^{--}] \quad (12)$$

$$[H^+] = [OH^-] = 1.0 \times 10^{-7} \quad (13)$$

combining (11) and (12),

$$[Hg(OH)_2] = [HgS_2^{--}] = {}^1/_2S \quad (14)$$

(13) makes things very easy. From (3), with $[H^+] = 1.0 \times 10^{-7}$,

$$\frac{[Hg(OH)_2]}{[HgOH^+]} = 1.4 \times 10^4 \quad (15)$$

From (2),

$$\frac{[HgOH^+]}{[Hg^{++}]} = 3.2 \times 10^4 \quad (16)$$

From (7),

$$\frac{[HgS_2^{--}]}{[Hg(SH)_2]} = 1.0 \times 10^7 \quad (17)$$

From (6),

$$\frac{[Hg(SH)_2]}{[H_2S]} = 6.3 \times 10^{-7} \quad (18)$$

multiplying (15) by (16) gives, using (14)

$$[Hg^{++}] = \frac{[Hg(OH)_2]}{(1.4 \times 10^4)(3.2 \times 10^4)} = \frac{S}{9 \times 10^8} \quad (19)$$

Multiplying (17) by (18) gives

$$[HgS_2^{--}] = 6.3\,[H_2S] \quad (20)$$

From (4),

$$\frac{[HS^-]}{[S^{--}]} = 8.0 \times 10^5 \quad (21)$$

From (5),

$$\frac{[H_2S]}{[HS^-]} = 1.0 \quad (22)$$

combining (20), (21), (22), and (14) gives

$$[S^{--}] = \frac{S}{1.0 \times 10^7} \quad (23)$$

Finally, using (19) and (23) in (1), gives

$$[Hg^{++}][S^{--}] = \frac{S^2}{(9 \times 10^8)(1 \times 10^7)} = 1.0 \times 10^{-51}$$

$$S = 3.0 \times 10^{-18}$$

Compare this result with 3.2×10^{-26}, which is obtained using the conventional approximation. Hydrolysis of anion and cation, and the formation of the HgS_2^{--} complex has increased the solubility by a factor of 10^8. Substituting back in the various equations above, the concentrations of all the species can be obtained.

$$[H^+] = [OH^-] = 1.0 \times 10^{-7}$$

$$[Hg(OH)_2] = [HgS_2^{--}] = 1.5 \times 10^{-18}$$

$$[H_2S] = [HS^-] = 2.4 \times 10^{-19}$$

$$[HgOH^+] = 1.1 \times 10^{-22}$$

$$[Hg(SH)_2] = 1.5 \times 10^{-25}$$

$$[S^{--}] = 3.0 \times 10^{-25}$$

$$[Hg^{++}] = 3.3 \times 10^{-27}$$

From this list we can see that $[Hg^{++}]$ and $[S^{--}]$ have in fact the *smallest* concentrations of all, so small that it is better to think of HgS dissolving according to the reaction

$$2HgS + 2\,H_2O \rightleftharpoons HgS_2^{--} + Hg(OH)_2 + 2H^+$$

and that Hg^{++} and S^{--} are formed by dissociation of these species. It must be made clear, however, that the solubility equilibrium constant K_1 is still correct: It is determined by potentiometric measurements of $[Hg^{++}]$ in solutions of known sulfide strength. What is incorrect is the assumption that $[Hg^{++}] = [S^{--}] = S$.

Example 5: MnS

Calculate the solubility of MnS in pure water and the concentrations of all species present. The equilibria are

$$MnS(s) \rightleftharpoons Mn^{++} + S^{--} \qquad K_1 = 2.5 \times 10^{-10}$$

$$Mn^{++} + OH^- \rightleftharpoons MnOH^+ \qquad K_2 = 2.5 \times 10^3$$

$$Mn(OH)_2(s) \rightleftharpoons Mn^{++} + 2OH^- \qquad K_3 = 1.6 \times 10^{-13}$$

$$Mn(OH)_2(s) + OH^- \rightleftharpoons Mn(OH)_3^- \qquad K_4 = 1.0 \times 10^{-5}$$

together with the water and H_2S equilibria quoted in Example 4. The calculation here is similar to that of Example 4, but since the dissolved species are present in greater amounts than H^+ and OH^-, the mathematics is much more complicated and space does not permit a detailed calculation. The results are as follows: MnS dissolves to an extent of 1.9×10^{-3} moles per liter, but hydrolysis is so extensive that $Mn(OH)_2$ precipitates to an extent of 9.5×10^{-4} moles per liter. The net solubility is 9.6×10^{-4} moles per liter, which is 60 times as large as the value (1.6×10^{-5}) calculated using the conventional approximation.

The concentrations of species in solution are:

$$[HS^-] = 1.9 \times 10^{-3}$$

$$[Mn^{++}] = 9.6 \times 10^{-4}$$

$$[MnOH^+] = 3.1 \times 10^{-5}$$

$$[H_2S] = 1.5 \times 10^{-5}$$

$$[OH^-] = 1.3 \times 10^{-5}$$

$$[S^{--}] = 3.0 \times 10^{-7}$$

$$[H^+] = 7.7 \times 10^{-10}$$

$$[Mn(OH)_3^-] = 1.3 \times 10^{-10}$$

For carbonates and phosphates of metals that hydrolyze only slightly the conventional approximation may yield solubilities which are within a factor of 5 or 10 of the correct value, but with metals like Mn^{++}, Hg^{++}, and Fe^{+3} where cation hydrolysis is extensive, errors may be several orders of magnitude.

The common ion effect calculation suffers more from complex formation than anything else, and in some cases (e.g. HgI_2 and KI) complex formation may be the principal reaction. Except for CuCl and the silver halides, the compounds listed in Table 1 show only slight complex formation.

Finally, a word must be said about the errors introduced by neglecting the electrostatic interactions of ions. The Debye-Hückel limiting law[4] can be applied to correct for this type of non-ideality at concentrations below 0.01 *M*. The corrections may be as large as 30% at $10^{-4}M$ if polyvalent ions are present. In the common-ion-effect calculations, where one ion is present higher concentrations than in a solution of the pure salt, the errors may be much larger, but the corrections above 0.01 *M* are largely empirical and data may not be available. These corrections usually are less than a factor of five (unless large concentrations of polyvalent ions are present), whereas neglect of chemical reactions that take place may result in an error of as much as a factor of 10^8.

In summary, the conventional solubility calculations are sometimes reasonably good, but more often may give very erroneous answers if important hydrolysis and complex formation reactions are neglected.

[4] HARNED, H. S., AND OWEN, B. B., "The Physical Chemistry of Electrolyte Solutions," 3rd ed., Reinhold Publishing Corp., New York, 1958.

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Programmed Instruction in Chemistry

An invitation to participate

Auto-instructional methods which sometimes use mechanical devices incorrectly called teaching machines[2] are based on the technique used by Socrates (*1*). The matter to be learned is presented in an orderly way, from simple to complex, from what is known to what is to be known, in a manner which insures that the student will actively attend to the presentation. If the student is momentarily distracted, instruction ceases, and resumes only when his attention is again directed to the exposition. The new aspect of Socrates' technique lies in its adaptation to large numbers of students, without the necessity for the actual presence of a teacher.

When this technique is used, the elementary instructional content of the classroom lecture can be decreased and the topic of the moment can be treated in in greater depth and made more interesting, thus encouraging students to learn more on their own, outside of class. Additionally, because the students are better prepared, those who do have difficulties can be helped more efficiently in person-to-person conferences with their teacher. As a result, the attention given to the individual student who needs help is increased in quality, with less expenditure of time. This article has been prepared to encourage the participation of other chemistry teachers in the preparation

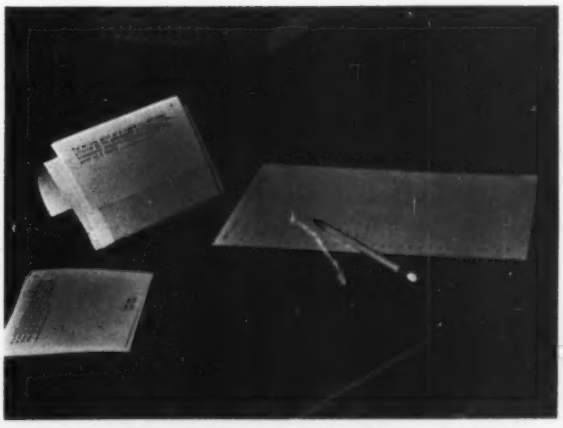

Figure 1. An easily made tutorial instrument (teaching machine).

[1] Prepared in connection with a research project at Earlham College supported in part by a grant from the U.S. Office of Education, Dept. of Health, Education and Welfare (Grant No. 7-12-026.00).

[2] Dr. John F. Baxter has suggested the term "tutorial instrument"; since this term more accurately describes the concept discussed here, it will be used in place of the less felicitous but currently popular term, "teaching machine."

of auto-instructional material and to stimulate its wider use in the instruction of students.

Day has discussed (*2*) the use of scrambled books in which the student selects a response from a given list of choices. The other major auto-instructional method requires the student to construct his own response. This is discussed[3] in detail here.

One of the simplest tutorial instruments, and one of the most satisfactory for initial use by a teacher who wishes to learn how to prepare auto-instructional material, can be constructed (*3*) from a sheet of stiff paper and a set of 4 × 6 in. index cards[4] (see Fig. 1). On each card a question, a statement, or other instruction to the student is printed. Each card in the set is numbered serially, and the packet of cards is placed, arranged in order, in the pocket on the front face. Usually the information on the first card will introduce the student to the topic to be considered on the underlying cards; and most of the cards will present questions to the student. The first question, on the first underlying card, is easy to answer. After studying this question, the student writes what he believes to be the correct answer on a piece of paper and then withdraws the card from the pocket. The correct answer is printed on the lower part of the card and is thereby revealed; the student compares this answer with his own response and then addresses his attention to the question on the next card, now uppermost in the packet of cards remaining in the pocket. Each question, and particularly its answer, leads to the question on the next card, in an ordered fashion. Instead of questions, if the orderly presentation of information requires it, some cards may carry statements or special instructions.

In some cases, a question may be correctly answered in more than one way. These several correct answers may be printed on the lower portion of the card; one of these should correspond to the answer written by the student. An answer, revealed by withdrawing the card from the pocket, can be followed by a statement which qualifies the answer or extends its significance beyond that indicated by the question to which it applies directly.

A complete series of statements, instructions, questions, and answers, arranged in order, is called a "program." One question from a program set up on index cards is illustrated in Figure 2.

Teachers who prepare a program for the first time are usually surprised by the results. The students learn little or nothing from the program. That is, the steps from one question or statement to the next are "too large." A program originally containing 20 discrete questions and statements must be altered so that 50–100 steps appear between the first item and the final conclusion. The almost universal reaction of teachers who have been through this regimen is the realization that before writing a successful program, they did not know how poorly they taught. Put another way, the teacher who learns how to write a program is in closer touch with the student at every step along the way and is therefore a better teacher in the classroom and laboratory.

The first draft of a program, on index cards, should be given to one or two students who then go through the program in the presence of the author so that he can obtain maximum benefit from this first evaluation of his work. During and after this first tryout, the program should be revised by removing some of the original cards, by adding new items, or both. Then it can be given to one or two more students and revised again. At this time the students can be given an examination to determine whether they have reached the objectives intended. After a further revision, the material can be reproduced by mimeograph or other means and the several papers stapled together. A few such programs are available upon request.[5] For a sample program, also see the article by Skinner (*4*).

In general, it is helpful to the student using a program if he is first told, by an introductory statement on the first card, what he can expect to learn from the program. Such an introduction should recall to the student some concept with which he is already familiar and briefly indicate that, with this as a starting point, he will learn about another concept. The new concept is then described in the introductory statement.

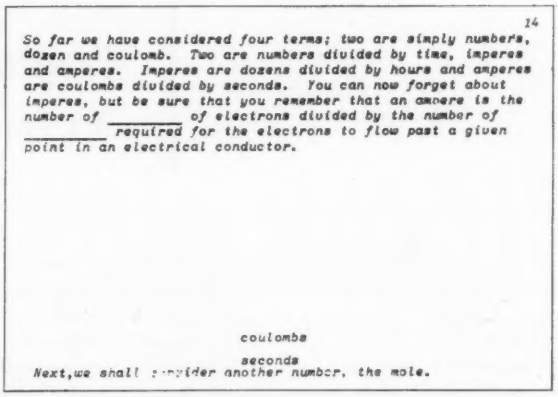
14
So far we have considered four terms; two are simply numbers, dozen and coulomb. Two are numbers divided by time, imperes and amperes. Imperes are dozens divided by hours and amperes are coulombs divided by seconds. You can now forget about imperes, but be sure that you remember that an ampere is the number of _____ of electrons divided by the number of _____ required for the electrons to flow past a given point in an electrical conductor.
coulombs
seconds
Next, we shall consider another number, the mole.

Figure 2. Sample card.

It is important to remember, in writing one's first program, that it is intended primarily to teach, not to test, the student. Hints and prompts that lead to the answer are legitimate and useful. For example, an item might read:

Just as an increase in temperature (other variables being held constant) causes an increase in the pressure of a gas, an increase in the quantity of gas confined in a container of fixed volume will cause an _____ in the pressure, when the temperature is held constant.

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[3] I wish to acknowledge the helpful criticisms of Professors O. T. Benfey and John A. Barlow, of the Departments of Chemistry and Psychology, respectively, at Earlham College during the preparation of this paper.

[4] A dimensioned sketch for the construction of this instrument from a manila folder will be supplied upon request.

[5] Professor O. T. Benfey, Earlham College, has programs for these topics: The Geneva System of Nomenclature for Alkanes; The Geneva System of Nomenclature for Alkenes, Alkynes, Dienes, and Cyclic Hydrocarbons. These programs are available from the author: Logarithms (a review); The Potentiometer; Electrolysis, Faraday's Laws, and Numerical Problems; Glass Blowing (end to end, tee, and ring seal); The Barometer and the Weight of the Atmosphere; How to Set and Read a Mercury Barometer; Vapor Pressure; The Boiling of Liquids.

As in any person-to-person discussion between teacher and student, a program should contain items which review or expand the concepts learned from prior items in the program. These can vary from simple review questions to statements which require that the student apply information learned from a prior item while new information is presented in the item at hand. Or, differences between facts or principles that have been presented, and qualifications of the implications that might be drawn from facts or principles, previously discussed, can also be treated in items which review or expand previously learned information.

Often, in writing a program, it is necessary to introduce a new subordinate topic. These should not be introduced abruptly. A transitional statement, indicating that a new subject is to be considered, and why, is usually necessary.

These, and other helpful suggestions on program writing are discussed more fully in two papers in Lumsdaine and Glaser (*5*). (The other papers and abstracts in this excellent reference also will be found useful to those interested in auto-instructional methods.)

As stated above, most beginners who write their first program find that they have not included enough separate steps in their program. Upon revision, the question is: How small shall the individual steps be? There is no clear answer yet known. If the steps are very small, no doubt the program will be clear, but it will also probably be boring to some student. One answer to this unsolved problem is to strive to word each item, no matter how small or large the "distance" from the preceding item, so that a student can realize a sense of accomplishment when he constructs his own response. Edwards (who favors the use of small steps) has suggested that each item in a program should be written in a manner which maximizes active participation by the student and minimizes exposition by the program writer (*6*). It may be true that this criterion is more important than either the criterion emphasizing a sense of accomplishment or the criterion based on the distance between items.

There are other answers to this question. Some have found it desirable to write three programs to cover the same objectives: one with large distances between items, one with intermediate distances, and one with small distances. This procedure is no doubt satisfactory, but it requires considerably more work. Another alternative is to use a "branching" program. Depending upon his answer to a question (usually this will be a multiple choice question) or upon his own estimate of the difficulty he is encountering, the student is directed to go next to item "Q" instead of item "R." Item "Q" then leads the student through a series of small steps, ending finally at item "S." The other alternative, item "R," goes directly, in one step, from "R" to "S."

As another alternative, a program can be written only for the moderately rapid learners; these individuals, in turn, are then paired with slower learners while the slower learners work their way through the program. In the process, if they are asked to do so, the better students will discuss the points that are difficult for the slower students at the time they are presented by the program, acting as apprentice Socratic teachers, to the benefit of both.

To many, one of the most cogent objections to the use of a program as a teaching tool is that students who are taught by this method will not learn to read an ordinary text or reference book with understanding. There is some evidence to contradict this conclusion, but it is probably also desirable (and there is no reason to exclude this procedure) to insert instructions at appropiate places in the program which direct the student to refer to a specific place in a specific book, or to find and study a reference (for example, in THIS JOURNAL or other suitable work, as an encyclopedia or reference book) which is only vaguely identified. The next item in the program itself would, then, refer to the material learned in such an excursion.

Conclusions and Recommendations

By using several different programs, each treating a particular topic, and requiring that these be studied before class as an adjunct to the material assigned from the text, a chemistry teacher can treat these same topics more fully in his lectures, or he can use the now available class time for a discussion of new topics which should be added to the curriculum.

Less time will be used outside of class to help students who find it difficult to understand the topics being presented, and fewer students will need such help.

However, a great deal of time is required to prepare, to test, and to properly revise a program. One teacher, working alone, cannot easily take full advantage of this method.

Those who are interested in developing programs in the field of chemistry are invited to communicate with the author who will attempt to set up a clearing house of information in this field. A brief descriptive statement listing the major objectives that the program is intended to meet, and a general statement describing the content and mentioning unique points,[6] should be furnished (less than one page of typescript should suffice). These will be reproduced and distributed to all who cooperate, for their information. As finished programs are developed, this author will, if informed, advise all other participants of the availability of such works.

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(2) DAY, J. H., J. CHEM. EDUC., **36**, 591 (1959).
(3) ALEXANDER, H., AND GILPIN, J., private communication.
(4) SKINNER, B. F., *Science*, **128**, 969 (1958).
(5) LUMSDAINE, A. A., AND GLASER, R., editors, "Teaching Machines and Programmed Learning: A Source Book," Dept. of Audio Visual Instruction, Natl. Educ. Assn., Washington, D. C., **1960**, pp. 401–15, 486–96.
(6) EDWARDS, W., unpublished work for the USAF, cited in FRY, E. B., BRYAN, G. S., AND RIGNEY, J. W., "Teaching Machines: An Annotated Bibliography," *Audiovisual Communication Review*, **8**, No. 2, 35 (1960).

[6] For example, in a program discussing some aspect of acidity, the choice of H^+ or of H_3O^+ should be stated; or, in a program on the solution of gas law problems, the use of $PV = nRT$, of $PV/T = P'V'/T'$, or other appropriate equation, should be indicated.

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Coordination Chemistry in General Chemistry Texts

In an article published in THIS JOURNAL in March of 1958,[1] Kirschner pointed out that he notes "almost complete exclusion of the topic of inorganic coordination compounds" in general chemistry courses offered in the colleges and universities in the United States. He goes on to suggest three basic postulates of Werner's coordination theory that might profitably be discussed in a course at the first year college level. After suggesting a definition of "inorganic coordination compounds," he points out that there are several appropriate subject areas into which coordination chemistry might be integrated. Thus he mentions topics such as water softening, the photographic process, the Mond process for nickel purification, amphoterism, equilibrium, biochemistry, and the chemistry of the transition elements or of nitrogen and phosphorus or of gold and silver.

In 1960, in THIS JOURNAL, Summers[2] suggested that high school chemistry texts should also be considered out-of-date if they do not include "Stock" nomenclature, atomic molecular, and ionic geometry, and complex ions, among other topics. Indeed, since complex ions and coordination compounds are definitely encountered in many courses after the "general" one, it would certainly seem highly desirable if not mandatory that the advancing student become acquainted with this broad area of chemistry.

Since 1958 many new texts aimed at the college first year course in chemistry have been published. One does not know to what extent these authors were influenced by the Kirschner article, nor is it particularly important to know this. Questions of concern to teachers in this field are first, whether the criticism was a valid one in 1958; and second, whether anything has been done about it in the textbooks.

It shall be the purpose of this paper to offer an answer to each of these questions on the basis of a survey of 36 college chemistry text books, 16 of which were published (but not necessarily written) after the Kirschner article. The books surveyed do not, unfortunately, constitute an exhaustive survey. They were chosen on the basis of availability among our colleagues at this university. We feel, however, that they constitute a representative sample.[3] All of the books were reviewed with the following questions in mind:

How does the text define "complex ion" and/or "coordination compound"?

Under what chapter titles is this done?

Does the text discuss the history of coordination theory at all?

Does the text discuss the nomenclature of complex ions, and does it use Stock nomenclature for other ions?

Is the Lewis concept of acids and bases discussed?

The answers to these questions, and the perspective gained in searching for them, form the basis for the following discussions and conclusions.

Definitions

In looking for definitions of *complex ion* and *coordination compounds* a fundamental difficulty of this "textbook research" soon became apparent. Essentially the difficulty seems to be that some texts, although discussing complex ions and related topics, never formally define the term. Other authors have apparently felt that a definition beginning, "A complex ion is .....," obviates the necessity for any further amplification of the topic. It is largely a matter of opinion as to which author has done the student a greater service. One must not conclude that the lack of a formal definition of these words condemns the book in any way.

Regardless of the interpretation to be placed upon the presence or absence of definitions, it is extremely interesting but perhaps not surprising to find that the words "complex ion" mean many things to many authors, and that there are almost as many different definitions as there are authors. These range all the way from, "Complex ions are ions containing two or more atoms" (Selwood), to the following discussion (Sanderson).

Chemical equations are usually written as though positive

EDITOR'S NOTE: Publication of this type of article is an important service which THIS JOURNAL can provide. At the same time we feel that it is equally important to remind readers that along with facts, this article expresses opinions. These are the opinions of the authors and publication should not imply endorsement in detail by the Division of Chemical Education of the ACS. This paper has not been refereed and has been edited by deleting specific references which might have embarrassed textbook authors. The authors of this paper will provide these references to interested readers.

[1] KIRSCHNER, S., J. CHEM. EDUC., **35**, 139 (1958).

[2] SUMMERS, D. B., J. CHEM. EDUC., **37**, 263 (1960).

[3] Books identified by author and year of publication are: Jones (1947); Pauling (1948); Mack, Garrett, Haskins, and Verhoek (1949); Barksdale (1950); Selwood (1950); Felsing and Watt (1951); Hopkins and Bailar (1951); Babor and Lehrman (1952); Babor (1953); Sanderson (1954); Sneed, Maynard, and Brasted (1954); Ritter (1955); Steiner and Campbell (1955); Sorum (1955); Scarlett (1956); Pauling (1956); Wood and Keenan (1957); Sienko and Plane (1957); Laubengayer (1957); Clapp (1957); Compton (1958); Frey (1958); Schwenck and Martin (1958); Gregg (1958); Watt (1958); Quagliano (1958); King and Caldwell (1959); Graham and Cragg (1959); Sisler, Vander Werf, and Davidson (1959); Baxter and Steiner (1959); Hutchinson (1959); Luder, Vernon, and Zuffanti (1959); Nebergall and Schmidt (1959); Hiller and Herber (1960); Taylor (1960); Ehret (1960).

ions existed as separate particles. Actually, however, this cannot be so, because, in any ordinary environment, positive ions will attract strongly the outermost electrons belonging to other ions or atoms or molecules. Polar molecules, such as those of water, are attracted by the positive ion. The result is that positive ions invariably are at least partly surrounded, and in effect partly neutralized, by either solvent molecules or other molecules, atoms, or ions. The forces of attraction are sufficient to require considerable energy to overcome them. They range from an ion-dipole electrostatic interaction to the actual formation of covalent bonds in which both electrons are supplied by the ion or molecule attracted to the ion. In the former case, the number of attracted particles is usually indefinite or constantly changing, and the stability of the aggregate formed is so slight that aggregates of definite composition cannot be isolated. In the latter case the number of attracted particles is usually quite definite, sometimes 2 and most frequently either 4 or 6, and definite aggregates are stable enough to be isolated or at least identified as such.

Such aggregates are called *complexes*, and when they bear a charge are called *complex ions*.

Of the 36 books only seven do not have what might be called a formal definition of "complex ion." Some of these write equations using complex ions in the course of the descriptive chapters with no comment on the bonding forces involved. One book defines "molecular compounds" instead. At least two very recent texts intended for rigorous courses never define a complex satisfactorily, and one of these seems entirely innocent of the existence of coordination chemistry as a part of the science.

It is interesting to note that the oldest and the newest books reviewed both offer good definitions. There seemed to be no correlation between the age of the book and the excellence of the definition. The oldest text in the survey has a very satisfactory three pages on coordination compounds and complex ions. If a chronological trend is to be observed, it is a trend toward more dissatisfaction with the "complex ion is a polyatomic ion" concept, and more attempts to define complex ion to include only complexes in the Werner sense. Of the 29 texts attempting a definition, only three of these then qualify their definition in some way to make the distinction mentioned above. Six, out of the 36, attempt to leave sulfate and similar common radicals out of the category of complex ions in the usual sense.

A popular way to avoid the "polyatomic ion" definition seems to be to base the definition on coordinate bond formation as a criterion. This, in fact, is what Kirschner has suggested:[1]

Coordination compounds: Compounds formed by the combination of two or more substances, each capable of independent existence, by means of coordinate covalent bonds.

This definition, which would include $H_3N:BF_3$, could not include the analogous $H_3N:BH_3$ since BH_3 does not have an independent (stable) existence. Furthermore, the student has little information at this stage to judge whether or not various species may have an independent existence.

Except for the phrase "each capable of independent existence," this definition then would say in effect, that if we recognize a Lewis acid-base interaction as the mechanism of the compound formation, we recognize that the compound contains a complex ion. The difficulty with this general approach, which leans on the coordinate covalent bond, is that current theories of the bonding in these compounds indicate that the bond is not this simple.[4] Also it presumes a knowledge of the mechanism of formation of the species involved, since coordinate bonds, once formed, are simply covalent bonds.

Three of the texts were cautious about the exact nature of the bond, the best of these having already been quoted earlier. Another of these includes sulfate ion as an example, and returns essentially to the "polyatomic ion" concept. The third, although including a discussion of bonding forces in complex ions, neglects to define them and seems to use "complex ion" as "complicated ion."

Two of the texts surveyed have definitions which, to us, seem confusing. One states:

The less active metals, while forming compounds with highly electronegative groups such as fluoride ion, frequently are bonded covalently, especially in their higher oxidation states, to give *molecular substances* or *complex ions*, such as $SnCl_4$, MnO_4^-, and $Cu(NH_3)_4^{-2}$ (sic).

The other, a new text, heads the confusing paragraph "Complex Ions and Molecules; Coordination Compounds." The author then discusses the formation of ammonium chloride from HCl and NH_3 and concludes:

. . . Since the compound ammonium chloride was formed by the combination of two simple stable compounds, it may be called a *complex compound;* the ammonium ion is called a *complex ion*.

From this one would infer that all compound-compound addition reactions yield a "complex compound" which is composed of "complex ions," an inference which does not seem satisfactory.

Finally, there seem to be several authors who tread a fine line between saying "polyatomic ion," and stating a coordinate-covalent mechanism; a prime example of this approach is:

When a simple ion combines with one or more other ions or with one or more neutral molecules to form a new ion, this new ion is called a *complex ion*.

Despite the apparent ingenuity of this definition, it reduces to something very similar to a "polyatomic ion" in a case such as this:

$$O^{-2} + H_2O \rightarrow 2OH^-$$

in which a simple ion (the student is familiar with the metal oxides and the oxide ion) combines with a neutral molecule to form a new ion (strictly two new ions, but hardly a complex ion). Or perhaps the definition will call to the student's mind reactions such as:

$$I^- + I_2 \rightarrow I_3^-$$

from which he would conclude that the I_3^- ion is a complex ion. Perhaps it is, but if so, should the definition of complex ion be so all-inclusive?

Location

Kirschner[1] described at some length the "appropriate subject areas" into which coordination chemistry might be presented quite conveniently, as mentioned earlier in this paper. The texts surveyed here were found to introduce the topic in several different ways. These

[4] SUTTON, L. E., J. CHEM. EDUC., **37**, 498 (1960).

are listed in Table 1 in the order of their frequency of occurrence.

Once again, caution is urged in interpeting these data. Table 1 lists the subject matter of the chapter under which the definition of "complex ion" is found, and in most cases this corresponds to the location of subsequent discussion of the topic. In some cases, though, several chapters should have been listed to indicate all the loci of discussions of complex ions. Furthermore, the discussion of complex ions is not synonymous with a discussion of Werner's coordination theory, which is perhaps what Kirschner had in mind.

Table 1. Subjects of Chapters Introducing the Topic of Complex Ion or Coordination Chemistry

Subject	No. of texts introducing topic here
Equilibria	6
Chemical bonding	5
Transition elements	5
Copper, silver, and gold	5
Separate chapter	4
Metals	3
Solutions	2
Acids and bases	2
Nitrogen family	1
Groups I, II, and III	1

Only in the case of a separate chapter is there no doubt about the presence of a thorough coverage of the topic. The "4" listed under separate chapter is perhaps misleading, because two of them are Pauling (the old and the new edition). The other two are Quagliano, and Baxter and Steiner. In all four of these texts the chapters are excellent, a strong recommendation for the separate chapter in all subsequent texts.

Table 1 also shows that the most popular location is under "equilibria," probably because these authors found the effect of complex formation on equilibria more unavoidable than the effect that the existence of these compounds exerts on bonding theories. To a large extent the introduction of complex ions under "solutions" amounts to an "equilibrium" approach. The same is true of "acids and bases," showing that the chapter titles do not invariably indicate the context of the discussion in an unambiguous way.

History

Only 11 of the 36 texts mention Alfred Werner. In some cases this is about all that is done. In other cases there is an adequate review of Werner's contribution to coordination theory. Even recognizing the trend toward less history and more theory in texts of this kind does not entirely excuse the omission of this great chemist's name from the vocabulary of the students. The omission is common, however, and some of the 25 books which do not mention Werner are satisfactory in other respects. It is our opinion that Hiller and Herber have a satisfactory paragraph devoted to Werner, and that something on this order is desirable regardless of the non-historicial approach used.

Nomenclature

Eight of the 36 books offer explanations of the nomenclature of coordination compounds, and two of these do not use modern nomenclature. About 16 texts explain or use the Stock system of nomenclature. One text assumes that the student will have had an above-average high school chemistry course and therefore does not present a section or even a paragraph on systematic inorganic nomenclature; in fact, it often uses formulas in sentences rather than names.

Nineteen of the texts do not mention Stock nomenclature or the nomenclature of complex ions. In general, though, the modern system is being introduced along with the old in most of the recent books. Perhaps the next generation of textbook writers will relegate the "-ous, -ic" nomenclature to an historical footnote where we believe it belongs.

Lewis Acid-Base Theory

Since coordinate bond formation results from a Lewis acid-base reaction, it is of some interest to notice how this acid-base concept is faring in our general chemistry texts. The Bronsted-Lowry concept is now firmly established in most recent texts, and the Lewis concept is perhaps too general to be of much help in giving a student the current chemical connotation of the words "acid" and "base." However, the Lewis concept is more and more being introduced in the general text, as Table 2 clearly shows. It is interesting to note that this follows the pattern set by Pauling in 1948.

Table 2. Occurrence of Lewis Acid-Base Concept in General Chemistry Texts

Date of publication	Lewis concept		Date of publication	Lewis concept	
	Have	Do not have		Have	Do not have
1947	...	1	54	1	1
48	1	...	55	1	2
49	...	1	56	1	1
1950	...	2	57	3	1
51	...	2	58	4	2
52	...	1	59	5	2
53	...	1	1960	3	...
			Totals	19	17

Summary and Conclusions

The introduction of coordination chemistry into general chemistry texts has been, like the recognition of these compounds historically, a slow and gradual process. The coordinate covalent bond and some simple ammine complexes found their way into most texts by 1948. As the general chemistry course content is the result of a continuous evolutionary process, thanks to the many willing authors, the pages devoted to complex ion formation, definition, naming and explaining soon grew to their present magnitude. Baxter and Steiner contains probably the best and most extensive coverage of coordination chemistry to be found in a text of this type, and should inspire even writers of "less rigorous" texts to include some of this material.

As for the validity of Kirschner's complaint, it is clear that an extensive presentation, such as that just mentioned, was rare before 1958. However, Jones' text in 1947 and Pauling's first edition are outstanding exceptions to this statement. Also apparent after this survey is the fact that a discussion of complex

ions is not rare in pre-1958 texts if one is willing accept this as "inorganic coordination chemistry."

Although inclusion of coordination compounds into Kirschner's "appropriate subject areas" would be desirable to a certain extent, it is our opinion that this is not the best solution to the problem. Instead we would prefer to see the possibilities of coordination foreshadowed early in a text as the student is made to realize that solution chemistry may involve more complicated participation of the surrounding molecules than the simple reactions he writes would indicate. Then, when types of bonding are discussed, a frank recognition of auxiliary valence possibilities should be made a part of the student's mental picture of ions in solution, particularly metallic ions. Finally, because of the large amount of current interest and research in the rapidly expanding field of coordination chemistry, we would prefer to see a separate chapter crystallizing these concepts with the aid of concrete examples of complex ions, evidence for ligand attachment, theories concerning the bonding, etc.

We would prefer to call SO_4^{-2}, NO_3^-, NH_4^+, MnO_4^-, CrO_4^{-2}, etc., polyatomic ions or radicals, but not complex ions. Complex ions we would define as the charged aggregates formed when a metal atom or ion becomes directly attached to a group of neutral molecules and/or ions (ligands). The complex ion resulting from this attachment may then react as a unit in subsequent ionic reactions and it also may show some tendency to dissociate or undergo substitution of its attached ligands. Coordination (or complex) compounds are then compounds which contain complex ions. Neutral complexes are a type of coordination compound resulting when the attached ligands exactly neutralize the ionic charge on the central metal atom.

Further comment on the evidence collected would go even further into the realm of personal preference We hope that this brief article will stimulate more discussion and ideas on the part of individual teachers of chemistry and textbook authors concerning the problem of how much coordination chemistry is warranted in the textbooks and in the general chemistry course.

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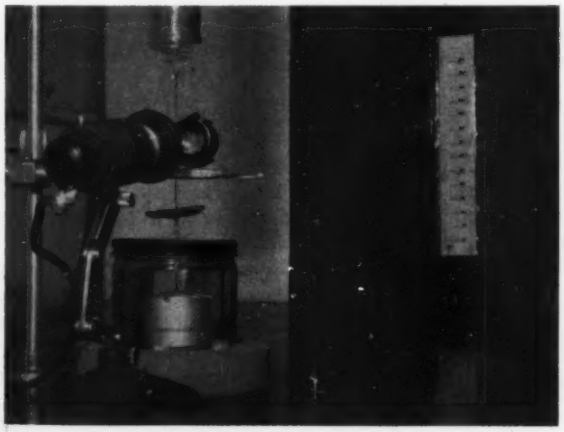
A Rapid, Direct-Reading Micro-Balance

The balance described here has proved useful for rapid, routine weighings in the range 0–130 mg where an accuracy of not more than ±0.25 mg is sufficient. It is easily and cheaply constructed, is direct-reading, and damps in about five seconds.

The photograph shows the essential parts of the balance. It consists of a "light" spring from a Jolly specific-gravity balance, a stiff suspension wire on which a cross-hair is mounted, pan, and oil damper. A sharply convergent beam of light from a microscope lamp is focused on the cross-hair, and the shadow is projected through a simple lens system onto a calibrated screen. A glass tube, 35 × 550 mm, protects the spring from air currents. The spring is hung from a cross bar sealed to the top of the glass tube, and a beaker inverted over the glass tube to exclude dirt. It is obvious that glass tube, damping pot, etc., must be rigidly fixed. Forceps are used to clamp the suspension wire when adding or removing a sample. The damper consists of an aluminum cylinder, open at both ends, made from a 35-mm Ansco color-film can; it is suspended in a light mineral oil. This arrangement gives maximum horizontal damping and minimum vertical damping. The lens was constructed from three simple hands lenses and gives a magnification at one meter of about 40 times; a condenser from a compound microscope could equally well be used.



The response of the balance is linear. Neither variation in room temperature nor heat given off by the microscope lamp significantly affect the performance. In several years use, the calibration has not changed. It is advisable to add and remove a 100 mg tare weight to the balance pan before starting a series of weighings. This presumably wets the damper suspension wire uniformly. The zero point can then be set by adjusting the scale vertically.

This application of ancient principles has proved useful to us; it may also to others. A similar balance with one-fifth the sensitivity and five times the range can be constructed from the "heavy" spring of the Jolly balance.

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Heat of Precipitation

A general chemistry experiment

Many freshman textbooks discuss the solution process of a solid electrolyte in terms of the ions in the crystal lattice interacting and hydrating with the water molecules and going into solution. A few textbooks give the more quantitative relationship

$$\begin{pmatrix}\text{Heat of}\\ \text{Solution}\end{pmatrix} = \begin{pmatrix}\text{Sum of Ion}\\ \text{Hydration}\\ \text{Energies}\end{pmatrix} - \begin{pmatrix}\text{Crystal}\\ \text{Lattice}\\ \text{Energy}\end{pmatrix}$$

Heats of solution are difficult to measure but for slightly soluble salts their converse, the heat of precipitation, can be easily measured.[1] Determining the heat of precipitation of two silver halides, and discussing the results, can be a simple but meaningful experiment for freshman chemistry.

The heat of precipitation experiment is carried out in a simple calorimeter constructed by each student from an Erlenmeyer flask, a beaker, and a −6 to 100°C thermometer graduated in 0.1°C. Considering the simple apparatus used, the results are quite good.

The Experiment

Construction of Calorimeter. Place a 250-ml Erlenmeyer flask in a 500-ml beaker. Pack the space between flask and beaker with wadded-up paper towels and wrap the beaker in a towel. Bore a hole for the thermometer in a rubber stopper that tightly fits the flask.

Heat Capacity of the Calorimeter. Pipet 100 ml of 0.25 *M* HCl into the Erlenmeyer flask. Put the thermometer in place and check the solution temperature until at two minute intervals the readings do not differ by 0.05°. From a graduated cylinder add 11 ml of 2.5 *M* NaOH solution. Replace the thermometer, read the temperature every 30 seconds and record the highest temperature that develops.

Assume that the heat of neutralization is 13,630 calories/mole. Calculate the heat liberated in the complete reaction of 100 ml of 0.25 *M* HCl Assume the solution weighs 111 grams and has a heat capacity of 1 calorie/gram. Calculate the calories absorbed by the solution and by the calorimeter per degree temperature rise.

Heat of Precipitation. Repeat the above procedure, first with 100 ml of 0.25 *M* $AgNO_3$ and 11 ml of 2.5 *M* NaCl, then with 100 ml. of 0.25 *M* $AgNO_3$ and 11 ml of 2.5 *M* NaBr solution. The temperature rise will be in the range of 3–4° C.

Note: All solutions are prepared well ahead of time and stored in the laboratory so they will be at room temperature.

The author will supply interested readers with a sheet indicating to the student a form for reporting results and outlining calculations.

Results and Discussion

Results of the first group to try this experiment are summarized in Table 1. The experiment has been used several times in somewhat larger classes with equally good results. Agreement of the class average value with the accepted value of the heat of precipitation is good.

The experimental results are discussed with the class. The heats of precipitation are arranged on the board in descending order. The average and the average deviation are calculated and compared with the accepted value as in Table 1. Students whose values come near the accepted value or at least fall within the range of the average deviation take considerable pride in their results. The poorer results are used to start an error discussion. Often carelessness in reading a volume or temperature or some simple arithmetic error is discovered in this discussion. The student's attention is directed to a critical evaluation of other errors. The facts that the density of the solutions is not exactly 1, that the solution heat capacities are not exactly 1, and that there is a heat-of-dilution effect are pointed out and discussed. Calorimetry in general can be discussed and more elaborate equipment and techniques can be described or even demonstrated if equipment is available.

Table 1. Heat of Precipitation[a] (cal/mole)

(Results of 12 groups of 2 students each, arranged in descending order.)

Heat of Precipitation[a] Calories/Mole		
Silver chloride	Silver bromide	
−19,640[b]	−25,660[b]	
−16,450	−22,070	
−16,100	−21,000	
−15,800	−20,700	
−15,600	−20,070	
−15,580	−20,000	
−15,520	−19,570	
−15,490	−19,490	
−15,450	−19,410	
−13,680	−19,080	
−13,300	−18,900	
− 9,220[b]	−14,400[b]	
−15,300 ± 700	−20,030 ± 750	Average
−15,650[c]	−20,190[c]	Literature

[a] Negative sign in thermodynamic sense of heat liberated. Most freshman texts give heat liberated a positive sign.

[b] Omitted from averages.

[c] Calculated from data in National Bureau of Standards Circular 500.

$$\Delta H_f\,[AgX\,(c)] - \Delta H_f\,(Ag^+, \text{hyp } 1\ M) - \Delta H_f\,(X^-, \text{hyp } 1\ M)$$

The results are further discussed as an exercise in thermochemistry. The fact that reversing the reaction reverses the sign of the heat effect is emphasized. Thus heat of solution = −heat of precipitation.

$Ag^+ + Cl^- = AgCl$ heat of precip. −15,650 cal/mole

$AgCl = Ag^+ + Cl^-$ heat of soln. 15,650 cal/mole

Presented before the Chemical Education Section, Southeastern Regional ACS Meeting, Birmingham, Alabama, November 1960.

[1] GURNEY, R. W., "Ionic Processes in Solution," McGraw-Hill Book Co., New York, 1953, p. 93.

Addition of reactions and associated heats to give the heat of a third reaction is demonstrated. The relative solubility of AgCl and AgBr is given and the student is asked to calculate the heat of the reaction:

$$AgCl + Br^- = AgBr + Cl^-$$

which is the difference $\Delta H_{ppt\ AgBr} - \Delta H_{ppt\ AgCl} = -4540$ cal/mole.

The results are also discussed in terms of the solubility process of vaporizing the ionic crystal to gaseous ions (crystal lattice energy) and hydrating the gaseous ions (hydration energy).[2]

$$\left.\begin{array}{l} M^+_{(g)} + X^-_{(g)} \rightarrow MX_{(s)} \\ M^+_{(g)} + H_2O \rightarrow M^+_{(aq)} \\ X^-_{(g)} + H_2O \rightarrow X^-_{(aq)} \end{array}\right\} \begin{array}{l} \text{crystal lattice energy, } U \\ \text{energy of hydration} \end{array}$$

Thus the heat of solution is the difference of two large negative heat effects:

$$\Delta H_{soln.} = \Sigma \Delta H_{hyd} - U$$

The student is given values of silver halide crystal lattice energies[3] and asked to combine them with his experimental results to calculate which ion (Cl^- or Br^-) has the larger hydration energy. Alternatively the student is sometimes given the hydration energies of the halide ions and asked to estimate which salt has the higher crystal lattice energy.

Related topics can be discussed with freshmen on very qualitative terms. For example, the contributions of Coulombic attractions, London attractions, and Born repulsions[3, 4] to the crystal lattice energy can be mentioned. Also the solubility mechanism of crystal vaporized to gaseous ions, then ions hydrated to form aqueous ions, leads directly to a discussion of the influence of solvent dielectric constant and the solvolysis reaction on solubility.

Discussing all the points outlined above would take much more time than most instructors could allow for one experiment. Each instructor can use his own discretion as to what subjects are important for discussion with his class. A brief discussion of errors and applications to thermochemical calculations would seem a good minimum of discussion for this experiment.

[2] LEUSSING, D. L., *in* "Treatise on Analytical Chemistry," edited by KOLTHOFF, I. M., AND ELVING, P. J., Interscience Encyclopedia, Inc., New York, 1959. Part I, vol. 1, Chap. 17, p. 707f.

[3] KETELAAR, J. A. A., "Chemical Constitution," 2nd ed., Elsevier Publishing Co., New York, 1958, Chap. 2.

[4] GOULD, E. S., "Inorganic Reaction and Structure," Henry Holt and Co., New York, 1955, p. 168 and Chap. 12.

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Analysis of Aspirin: A Conductometric Titration

Student interest often can be increased or even turned into enthusiasm by using other than the conventional reagents in an analytical experiment. We have found this to be true when students were asked to determine the amount of aspirin in a regular (5 grain) or children's size (1.25 grain) tablet. Total acidity can be assumed to be acetylsalicylic acid, formula weight 180.2. The figure shows typical student data obtained with a solution of one 5-grain tablet in 500 ml H_2O; $^1/_5$ aliquots were employed in a conductometric titration.

A Fisher Titrimeter can be used, but we have found that quite satisfactory results can be obtained by using instruments available in kit form which have been designed as resistance-capacitance testers to sell for about $20.00.[1] They operate on 60 cycle ac and use an electronic mill indicator. Although the potentiometer slide-wire in these is not uniformly linear, if conditions and cell constant are such that the middle two-thirds of the scale are used, errors are probably less than those inherent in graphical evaluation of the data.

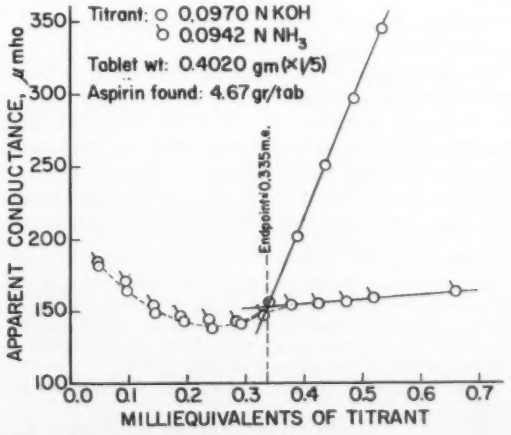

Figure 1. Direct titration of aspirin.

[1] Among other sources, instruments of the type described can be obtained from (a) Allied Radio Corporation, 100 N. Western Avenue, Chicago 80, Illinois, No. 83Y124 Knight-kit R/C tester; and (b) Heath Company, Benton Harbor, Michigan, Model C-3 Condenser Tester.

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Heat of Reaction and H_2SO_4 Concentration

A general chemistry experiment

The heat of a reaction is often observed but seldom measured in general chemistry work. Perhaps the most familiar example is that of the heat generated when sulfuric acid is added to water. The amount of heat evolved in this reaction must be related to the concentration of the acid used, and therefore the concentration of an acid sample can be determined quickly by reference to a concentration-heat of reaction curve derived from work with acids of known concentration.

Examination of the literature revealed very few previous attempts to do this. Howard[2] suggested a method for determining the concentration of at least 92% sulfuric acid and of oleum by measuring the temperature rise when definite quantities of the two were mixed in a Dewar flask. In this case the heat evolved was primarily that from the reaction of sulfur trioxide with water. Curtis and Miles[3] used essentially the same method for plant control testing, but preferred to use larger amounts of acid and oleum to measure these by volume. Richmond and Merreywether[4] used a thermometer reading to 0.01°C to measure the temperature rise on mixing 92–98% acids with water, and devised a formula with correction factors to calculate the concentrations of the acids.

We proposed to devise a simple, but fairly accurate, procedure for obtaining a measure of the heat of reaction between water and sulfuric acid of various concentrations, and to apply it to determine the concentration of unknown acid samples within the range of 50–98%.

Preliminary experiments showed that 5 ml of acid added to 25 ml of water gave temperature increases varying from about 11 degrees for 70% to 44 degrees for 98% acid. These effects seemed suitable for our measurements, and thereafter the volumes of acid and water were not changed.

The early quantitative work was done under the most favorable conditions. A pint-size Thermos bottle was used as a calorimeter. A number of acid samples were prepared, ranging from 40–98% in strength, and were standardized by titration. 5 ml of an acid was added to 25 ml of water in the bottle, and the temperature rise was measured with a 0.2°-thermometer. The temperature increases for the various samples were plotted against their concentrations. Then several acid samples of unknown strength were similarly used, and their concentrations determined by reference to this curve. The following results were typical:

Temp. rise, °C	% from curve	% by titration
7.7	63.5	63.8
14.9	75.4	75.9
30.4	89.8	90.5

Encouraged by these results, experiments were continued to simplify the equipment. If was found that equally good results were achieved by substituting for the Thermos bottle a small beaker wrapped in paper towelling, or surrounded with Vermiculite contained in a larger beaker. Further experiments were performed to determine the effect of the initial temperature of the water. Using 75.8% acid, and water at 13°, 21°, and 27°C, the temperature increases were 15.5°, 15.2°, and 14.8°, and the % acid 76.0, 75.7, and 75.3, respectively. These experiments indicated that good results can be expected by starting with water at room temperature.

Examination of the reference curve shows that a nearly linear relationship obtains in the region of 80–98% acid concentration. Within these limits, one degree rise in temperature is equivalent to 0.6% acid, and an ordinary thermometer graduated in degrees can be used without greatly reducing the accuracy of the determination. Below 80% acid the slope of the curve is less and is variable, the accuracy expected dropping to about 4% acid per degree rise between 50 and 60% acid concentration. These estimates were confirmed by class trials of the method given below, the students using ordinary 1° thermometers. Using two unknown acids of 63.6 and 82.6% strength, the class averages were 64.2 and 82.9%, with maximum deviations of 3.5 and 0.9%, respectively.

The Experiment

Insulate a 100 ml beaker by wrapping it with paper towelling. (Alternately, one may set the beaker in a larger one and surround it with Vermiculite or asbestos powder, or one may use a small Thermos bottle.) Suspend a thermometer in the beaker such that its bulb nearly touches the bottom of the beaker. Add exactly 25 ml of water from a buret or pipet, let it stand a minute or two, and record its temperature as accurately as possible. Measure exactly 5 ml of an acid of known strength in a pipet, hold the tip of the pipet below the water surface and let the acid flow into the

[1] This paper reports the work done by Leegwater and Vander Ploeg as part of their freshman work at Calvin College For other reports of this continuing program see THIS JOURNAL, **34**, 133 (1957), **35**, 412 (1958), **36**, 494 (1959), **37**, 137 (1960).

[2] HOWARD, H., *J. Soc. Chem. Ind.*, **29**, 3 (1910).

[3] CURTIS, R., AND MILES, F., *J. Soc. Chem. Ind.*, **39**, 64 (1920).

[4] RICHMOND, H., AND MERREYWETHER, J., *Analyst*, **42**, 273 (1917).

water while stirring with the pipet. Continue stirring until the temperature reaches a maximum and record this reading.

Rinse and dry the beaker and pipet, and repeat this procedure with other samples of acid whose strengths are known to lie within the range of 50–98%. Draw a curve plotting per cent acid as abscissa against the rise in temperature as ordinate. Then repeat the procedure with the acid samples of unknown concentration and determine their strengths by reference to the curve.

From the slope of the curve at the concentrations of the unknowns, and assuming one can read the thermometer to 0.2°, estimate the maximum accuracy one may expect in these determinations.

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Dye Solutions for the Preparation of Colored Slides

In the course of preparing illustrative material for technical presentations; it was found that more striking, clearer, cheaper, and easier-to-prepare slides could be made by dyeing black and white transparencies than by taking color photographs of original colored illustrations. In the latter method, not only must the original graphic material be prepared in color but the resulting transparencies generally lack brilliancy. In our approach we simply make negative transparencies, on film or glass, of the black and white illustration and then hand-tint the negative as desired. The resulting colors can be made quite brilliant. This technique can be used to differentiate individual curves on a complex graph or to identify fields or various portions of a slide for ease of referral during a lecture.

Since the colors are applied by hand, this is one limitation to the application of the method. No closer resolution can be made between adjacent colored regions than that permitted by the width of the applying device. We have used fine brushes and even single fibers, however, to differentiate as many as five crossing curves, with each possessing its individual color. This happy possibility exists because we deal with negative transparencies where the opaque regions permit "slopping-over" of the colors.

For the dye solutions we initially tried various water-base colored inks but these proved generally to give poor results. They either did not wet the slides, gave a poor film, or depended for their color on opaque pigments. The glass-marking inks were thick and difficult to apply, and proprietary dye solutions gave erratic results. In the long run it was found best to prepare our own solutions from commercially available dyes. Table 1 identifies the respective dyes, with the suitable solvent, color, and a quality rating (based on brilliance, uniformity of film, depth of color, and general ease of application). In some cases a drop of aqueous Aerosol OT (American Cyanamid Corporation) was added per 5 ml of dye solution to facilitate slide-wetting, but this proved generally unsatisfactory because of uncontrolled spreading on the slide.

All of these dyes are standard biological stains and are available at most laboratory supply houses, including Sargent, Matheson Coleman and Bell, Eastman Chemical, Fisher Scientific, Harleco, Mallinckrodt, and others. Dyes tried which proved less satisfactory as to solubility, color uniformity, or other significant properties include the following: Orange G, Congo Red, Alizarin Red S, Titan Yellow, Methylene Blue, Nigrosin, Alizarin Blue S, Methyl Orange, Methyl Green, *p*-dimethylaminobenzylidene Rhodanine, Quinalizarin, Phthalocyanine (metal-free) and Carmine.

In the use of the dyes recommended, practice is required to obtain a neat and uniformly colored slide but the results can be quite impressive.

Table 1

Dye	Color	Solution	Quality
Fluorescein (water soluble)	Yellow	1	E^+
Acridine red	Lavender	1	E^+
Methyl violet	Deep blue	1	E^+
Aniline blue	Light blue	2	E^+
Eriochrome cyanin R	Brown	1	E
Azure A	Blue	1	E
Neutral red	Brown	1	E
Acriflavine (neutral)	Yellow	2	E
Quinoline yellow	Light yellow	2	E
Alizarine yellow GG	Light yellow	2	E
Methyl red	Brown	1	G^+
Naphthol green B	Green	2	G^+

Solutions: 1, 500 mg dye dissolved in 5 ml anhydrous ethanol; 2, 500 mg dye dissolved in 5 ml ethanol—2 ml water.
Quality: E, excellent; G, good.

Proceedings of the

California Association of Chemistry Teachers

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New Fundamental Designs for Academic Chemistry Buildings

In the last two years a radical change in basic architectural design of college chemistry buildings has taken place, largely under pressure of building economics. Specifically, this means a nationwide interest in the central-utility-channel or "core" plan. Unfortunately, the use of such a system is largely outside the experience of two important groups, namely (*a*) academic personnel, and (*b*) various authorities who make safety regulations. In view of decided controversial opinion in these two groups, it is important that faculty building committees now hoping for new laboratories should give critical attention to the problem of the core design and not wait for a newly-appointed architect to give them a surprise.[1]

This presentation of the central-channel idea does not refer to the interior *shaft* plan, in which public services are brought up in a building through giant chimney-like passageways distributed at intervals through or around a building block. The shaft plan, in the present writer's opinion, is an inferior half-solution of the problem, at least outside the domain of skyscraper laboratories. It will therefore not be discussed here.

The present article makes no attempt to portray a whole building. Such a problem, with a multitude of options in location of entrances, stairways, elevators, windows, and other details, is the job of an architect experienced in laboratory design. Attention here is confined to the succession of spaces across a given building block or wing, depicted below in simplified sketches of representative portions of the structure.

Classical Design

Up to recent date, the conventional plan of Figure 1 was customary, placing laboratories, offices, etc., on either side of a standard corridor, which in the past sixty years has been gradually narrowed from about 15 feet to the present almost irreducible 8 feet in width. Just outside the corridor walls, on each side, an easement for flues and public utilities was at least vaguely designated; in 1950–55 this might have been about 18 inches wide, two years ago 24 inches, and today 30 inches. Widening of this easement comes from the continued demand for radically improved ventilation and air conditioning. It is thus the flue and pipe problem that has exhausted the patience of the architectural engineer, and is now moving the architect whole-heartedly into the central-utility-channel plan.

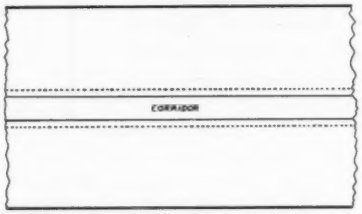

Figure 1. "Classical" arrangement.

One of the toughest assignments of this type was that handed by the Regents of the University of California to Risley and Gould, architects,[2] culminating in the recently-dedicated Harald Sverdrup Hall on the campus of the University's Scripps Institution of Oceanography at La Jolla, California. Nobody is quite sure just what will go on inside this laboratory during the next few years, required for development of the new School of Science and Engineering, University of California, San Diego, which will include the Scripps division. Extreme flexibility in use for physical and life sciences was the keynote in planning. The designers have accordingly made a radically new contribution to technical architecture.

Chemistry-building committeemen figuring out inspection itineraries may well put La Jolla on their lists. In the same excursion, still another core job may be conveniently visited at Corvallis, Oregon, in the new physics-chemistry structures of Oregon State University. Furthermore, the huge postwar population growth on the Pacific coast has produced several chemistry buildings of other patterns, thus offering opportunity for an excellent junket in the far west for the said committeemen. These projects include San Diego State College, UCLA (1952 building plus the large Chemistry Building Addition scheduled for construction in 1961–63), Occidental College, Gilbert Lewis Hall at Berkeley, the extensive and decidedly

[1] For the benefit of building planners, attention is called to the forthcoming comprehensive publication from the Committee on Design, Construction, and Equipment of Laboratories, Division of Chemistry and Chemical Technology, National Research Council. This new volume, edited by Harry F. Lewis, is entitled "Laboratory Planning for Chemistry and Chemical Engineering," and should be available from Reinhold Publishing Corporation, New York, by the end of 1961.

[2] 2502 West Third St., Los Angeles 57, Calif.

novel multistory Latimer Hall now under construction at Berkeley, and the new structure just completed for Washington State University at Pullman.

Central-Channel Plan

In place of the customary central corridor, a channel or noninflammable canyon passes lengthwise through the building, from roof to basement floor. Walls of this channel are normally of "two-hour" class, as referred to fire-control codes. Width of the channel, wall to wall, should be 12–16 feet, although some earlier plans, without close attention to air conditioning requirements, have included channels as narrow as 7 or 8 feet.

The proposal of 12 to 16 feet at first glance seems to be unwarranted extravagance, but such an assumption is not justified. A "core" building, after all, is nothing but two buildings back to back, separated by the channel. An increase in spacing from 8 to 12 or 16 means only a negligible increase in cost due to extra outer end walls, roof and basement floor—provided the requisite land is available. On the other hand, the wider space offers great convenience not only in contract installation, but also in opportunity for new mechanical installations required in the unforeseen research future. Fans, pumps, etc., are rapidly installed.

In summary, the core scheme, with wide spacing, promises at least 10% cut in the mechanical cost which might have been expected in a "classical" job. When the contractors get accustomed to bidding on the new designs, the discount of 10% may expand to 15 or even 20%. Most of this saving is in the really costly element, labor. For example, the contractor does not have to worry about pipes or flues from one floor threatening to conflict with a door above or below.

Instead of a conventional floor at each level, a steel grating catwalk, 30–36 inches wide, similar to equipment well known in the engine rooms of large ships, runs lengthwise in the channel. This catwalk rests on small I-beams spanning the channel at appropriate intervals. At La Jolla the catwalks are displaced to the side, as shown in Figures 2 and 3, allowing certain economies in grouping of pipe and flue risers, fans, etc., which occupy the core space. Room is still allowed between catwalk and wall for pipe runs. There is no reason, however, why a center position cannot be used if it suits the convenience of the architectural engineer and future maintenance staff. Figure 2 shows a cross section of one of the La Jolla building units in simplified sketch.

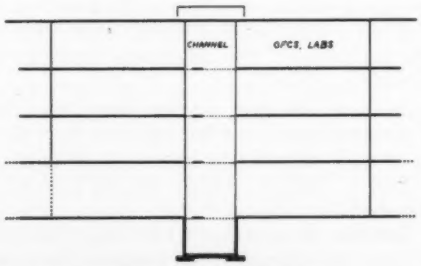

Figure 2. Cross section of a channel building.

Some architectural engineers prefer two catwalks, one on each side, with ventilation flues down the middle of the channel, which would require a relatively wide channel.

Grouping of Offices and Laboratories

Figure 3 shows a representative portion of one of the earlier proposals for a core laboratory building. In this structure it is assumed that the building is so long (east to west in the figure) that staff workers would object to the necessity of a long detour around the end of the building when they wish to go to the laboratory

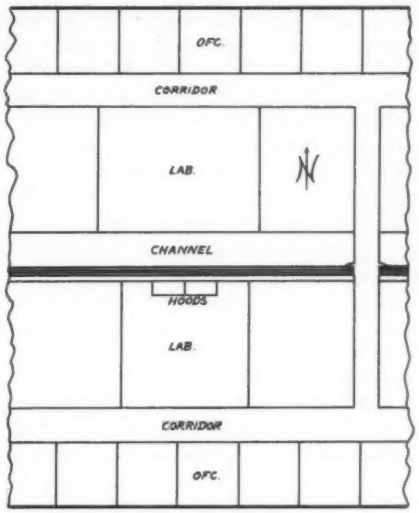

Figure 3. Channel building with cross-connecting subcorridor.

just across the channel. Accordingly, a cross subcorridor, virtually a rectangular fireproof tunnel about $7\frac{1}{2}$ feet high and 5 feet wide, is occasionally inserted. Such a subcorridor is shown at the right of Figure 3, connecting north and south main corridors. This tunnel is of ample height for pedestrian service, but still leaves room in the channel for a few horizontal pipe lines, east to west, if needed. There is no necessity for expanding it to permit transportation of large pieces of furniture, as one expects in a main corridor. Dimensions of the subcorridor should be cleared with code authorities at an early date.

One of the most conspicuous advantages of the core plan is seen in the location of hoods just outside the channel. Not only do such hoods have simple, relatively frictionless flue runs, but as centers of fire hazard they are happily located as far as possible from escape doors.

Figure 4, a composite of ideas from California, Illinois, and Oregon plans, shows the treatment of certain objections to Figure 3. Item A, for example, meets the criticism that the Figure 3 plan provides too many offices and minor rooms without mechanical equipment, and not enough laboratories. Since offices with south exposure are deplored, the lower row is deleted, leaving a porch or balcony in a mild climate, or a glassed-in corridor in a cold climate.

Item B implies that the "OFCS-LABS" areas are designed for movable partitions, and can be cut up to accommodate a variable proportion of office-laboratory schemes. Two balconies or exterior corridors are then

provided, and the special rows of offices (Fig. 3) omitted.

Item C solves the office-laboratory problem by direct replacement of the southerly bank of offices (Fig. 3) by a third row of laboratories. This scheme poses the problem of bringing public-utility services, flues, etc., to the new south row of laboratories. This is achieved by running cross tunnels, about 2 × 3 feet in cross section, under the ceiling of the middle row of laboratories; or some of the services may be introduced from below in the classical method of Figure 1. The plan is then virtually a hybrid of the classical and core systems; but the argument for the whole layout is somewhat weak.

Item D, apparently bordering on the ridiculous, may be significant on very costly urban terrain. It might be considered where it is critically important to house a large laboratory plant on a single city block. In summary, it is obvious that the architect can fill up a rectangular area of almost any large size and of any proportion by some special sequence of corridors, laboratories, and channels. Nor does the whole assembly have to be a complete rectangle.

In these assemblies, Figures 2 to 4, with interior corridors as narrow as 6 to $6^1/_2$ feet, it does now look as if the architects have reached the absolute minimum. Although two corridors or balconies may be required per floor, the yield of net assignable ("net useful") area in a chemistry building in ratio to gross area, in comparison with the classical arrangement, is fully up to an economical standard, provided the channel itself is appraised on a cost basis, and not on mere square or cubic footage.

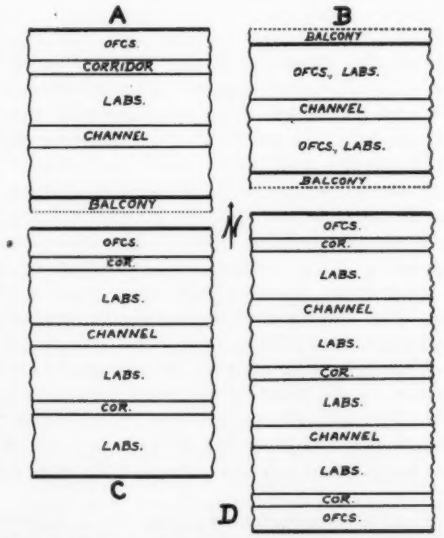

Figure 4. Miscellaneous office-laboratory-channel-balcony groupings.

Objections to the Channel Design

Sharp criticism of the core plan comes from many academic workers who object to windowless laboratories, and particularly to windowless offices. Choice of the Figure 4A plan, permitting desirable north windows, solves the office problem. The argument against windowless laboratories, however, is weakened by the almost universal practice of workers, even in sunny southern California, of using fluorescent lights day or night, summer or winter. But that is not a complete answer to the complainants.

It is not fair to brush aside the protest against windowless laboratories merely by provision of ample fluorescent illumination with attention only to foot candles. One must remember that the virtue of daylight (shaded) is not solely a matter of opportunity to see the birds and flowers. The illumination engineer should consider glare control—which means no direct rays from bare fluorescent tubes—and along with such a precaution make the proper prescription of tints of fluorescent light, plus an item often ignored, the best tints for laboratory walls. Only by proper treatment of all of these factors can one replace daylight with really satisfactory results.

In the face of reluctance of administrative authority in some institutions to provide adequate ventilation and air conditioning, the proponent of the core plan has a "left-handed" argument. Such forced ventilation is absolutely mandatory in a core structure, since one cannot ignore the problem with mere advice to "open the window!"

A minor objection—perhaps more than "minor" to the researcher whose office and laboratory work are very closely inter-related—is voiced against the Figure 3 or 4A plan, where one must cross a public corridor to go from office to laboratory. Figure 4B, with windowless laboratory and offices located so that they enjoy both daylight and artificial light, is then the most promising option. Removable partitions in such situation are favored, particularly in view of changes in demand for secretarial service in the future.

Fire Department Objections

Still more critical objections come from conservative writers of the codes for municipal and state fire and safety bureaus. For example, one major city authority in California will not approve a core building unless there is a complete, sealed-off, reinforced-concrete floor at each story level in the channel. Such drastic requirement completely invalidates the whole scheme.

Safety authorities insisting on these floor partitions in the channel argue first from their long-time requirement that multistory buildings must not have long vertical passageways which could behave as huge chimneys in a fire emergency. Such unimpeded passageways have long been outlawed in hotels and office buildings.

The same authorities are also fearful that a gas-air mixture, caused by leakage in a room not normally occupied by possible observers with sensitive noses, might explode. Such explosion in an undivided canyon might be disastrous. The objections are countered in the following manner:

(1) Exclude fuel gas service from the channel, placing the gas risers near corridors in the "classical" manner. Gas is of declining interest in modern laboratories anyway, and the added cost of pipe exclusion would be slight.

(2) Exclude all woodwork or other inflammable matter from the channel. All combustible insulation on wires, including tarred cloth, rubber, and plastic, would be covered in conduits, as already required in many city codes.

(3) Arrange the ventilation system so that air is

changed in the channel regularly, at least three times per hour. Such air need not be (costly) fresh air, but may be an offtake from habitable rooms near the channel, such as offices or classrooms.

(4) See that atmospheric pressure in flue lines (in the channel) is negative, thus forestalling leakage of an inflammable vapor into the channel from a hood. This specification would probably follow the normal engineering design of the hood system anyway.

Even though there are appropriate remedies for the fire hazards, the fact remains that certain "codes" are hostile to the channel plan, and there should be a very definite understanding, not requiring concrete-floor regulations, before any college administration encourages its architect to attempt a core building.

If one wishes to be really pessimistic, and still retain a practical channel structure, he may design roof hatches and louvered end wall sections which would yield to a gas explosion without great damage. A methane-air explosion after all is propulsive, not *brisant*, and the hazard is limited thereby.

Special Problems

The provision of ample space for flues in the central channel permits an unusual solution for the flue lining problem. First one may as well concede that there is no perfect flue-lining material. Even stainless steel, a costly material, has been proved in the University of California installations to have only a short life. A ceramic standpipe, too bulky for the classical building, might serve in a channel of ample width, but as of present date the sealing materials joining ceramic segments are too porous and absorptive to be acceptable to the growing number of researchers using radioactive materials.

One rather practical and realistic suggestion, adopted in the Risley-Gould job at La Jolla, is to use cheap black sheet iron, epoxy coated, and fabricated in standard units which are readily and cheaply replaceable. Elegance of appearance is obviously of no concern. Such sheet metal may cost only one fifth as much as stainless steel. Although replacement will be slightly more frequent than with stainless steel, the operations of renewal will be conducted in the channel where researchers will not be disturbed.

Penetration of the channel wall at various future dates could be a nuisance to researchers especially in walls of brick or concrete. A much better plan employs the approved "two-hour" design, under which the wall consists of light steel studdings, steel lath, and one inch of plaster. Little disturbance is foreseen in the future installation of a new piped service.

Acknowledgment

Acknowledgment is made of valuable technical assistance in the La Jolla design project by Mr. J. W. Tippetts, in charge of the La Jolla office of Architects and Engineers, University of California, and Admiral C. D. Wheelock, U.S.N. retd., naval designer and member of the Scripps Institution staff, who collaborated with Mr. Stanley R. Gould, executive architect.

Color Filmstrips of Atomic and Molecular Models

Filmstrip enthusiasts can point with pride to this series carefully designed by J. Arthur Campbell and beautifully executed by Encyclopaedia Brittanica Films. It is impossible to categorize a filmstrip as "good" or "poor." Even the best can have a poor effect if used by the teacher who has little understanding of their place in his arsenal of teaching aids. Furthermore, pretty pictures on film, even backed up by an "official" reading script, are no substitute for a teacher's knowing the subject thoroughly.

The present series is potentially an excellent teaching aid. The teacher who does not know his modern structural theory had better plan to spend some time with these films, recent textbooks, and a collection of Campbell's articles published in THIS JOURNAL before going into class with them the first time. These films are Art Campbell—all the way through. The thousands who have heard him, surrounded by wood, cork, and styrofoam in brilliant colors, always are impressed with the beautiful logic of it all, yet find it difficult to substantiate in detail. He is a master, both of his subject and of its visualization. Garden-variety familiarity with both, as possessed by most teachers, needs much more than these films and a projector to achieve effective teaching.

The following suggests in outline what the user will find. The number of frames given is the actual number of useful views, excluding titles, credits, etc. Note that these numbers vary greatly from the advertised "approximately 32 frames each." The price is $6.00 each, $42 for the set, available from Encyclopedia Brittanica Films, 1150 Wilmette Ave., Wilmette, Illinois.

9191 *Relative Sizes of Atoms* (25 frames). From the periodic table through visualized molar volumes to shortening of internuclear distances by multiple bonding.

9192 *Relative Sizes of Ions* (18 frames + 3 repeats). Octahedral and tetrahedral holes get filled by ions of appropriate size. The effect of size of the ions on alkali halide crystal form is elegantly portrayed.

9193 *Sizes and Shapes of Molecules* (26 frames). Excellent photographs of a great variety of models could have been greatly augmented by bond angle and dimension data. Why should the label PCl_5 be as big as the model? There is admirable use of contrast and sequence so that even as sophisticated a concept as the rotation of NH_4^+ ions in $NH_4^+Cl^-$ is clear.

9194 *Shapes and Properties of Molecules* (22 frames). Gradations in properties of binary hydrides and oxygen-containing acids are interpreted in terms of size effects. The $H_2 + O_2$ reaction mechanism is prettily modeled, but the size and shape correlations are tenuous.

9195 *Crystals and their Deformations* (20 frames). Campbell at his best, but the ideas are better than their execution. The actual crystals are dwarfed beside the models; KCl gets called NaCl; the deformation of close-packed spheres looks unconvincing. The demonstration of glide and cleavage planes is neat and clear.

9196 *Packing of Atoms in Crystals* (21 frames). The best strip of the set. Another whole conceptual dimension is used—the end view of 300 pieces of glass tubing to demonstrate regularities and irregularities in crystal growth. Superb!

9197 *Construction of Molecular Models* (47 frames). The complete do-it-yourself course. All the tricks of the trade for using a homemade lathe, jigs, and tools to turn the everpresent styrofoam into a room-full of molecular toys.

W. F. K.

Report of the New England Association of Chemistry Teachers

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Asking Students To Use Facts

For, the first decade of his career as a teacher, the author used a method of presentation that was essentially informative. He featured the descriptive fact and predeveloped idea, often at the expense of a thorough discussion of the basic concept. Occasionally he asked his students to use facts, but more often he expected only that his students remember them. His approach to numerical problems was based on the mastery of certain generalized types. During this period, the enjoyment gained from the classroom seemed ample, but during his second decade of service the author has uncovered a greater and more invigorating enthusiasm by using a different approach. He presents that approach here because he believes that all teachers are seeking a method of teaching which is genuinely more satisfying. He believes that all will be interested in a specific teacher's current personal doctrine. Although the approach is not new, it has particular merit because of its gradual evolution over the years, influenced strongly by many contacts with teachers and students.

The current pattern is designed to help the student to evaluate, digest, and use a selected body of chemical facts and concepts. Its major intent is to use chemistry as a means of enticing students to think and reason logically. Stress is placed on the application of facts, and especially on an understanding of the ways in which valid concepts may be applied to the solution of original problems. So-called descriptive chemistry, which provides the facts, cannot be neglected completely, but most of the class time is devoted to explanation and interpretation. In this way it is hoped that those students who forget the descriptive aspects of their chemistry course will at least retain the mental attitudes that they have learned to use there. Indeed, it is probable that the intellectural exercise which they have gained in such a course is more valuable to them than a cursory knowledge of the subject matter. Conversations with former students seem to verify this.

As taught to an introductory class of not less than 300 students, this approach appears to be different from any that many of them have encountered previously. As a consequence, their attitudes and grades cover a wide spectrum. Those who both expect and want to be educated realize that the use of facts and ideas in chemistry is a pleasant, stimulating experience. Those who expect and want only to be trained to follow specific procedures, and to be told just what to memorize and repeat, are usually quite disappointed. Fortunately, many of these latter finally change their points of view when they realize that their knowledge is more valuable once they have learned how to use it.

Examinations Are the Key

The grades earned in writing different kinds of examinations seem to indicate that most college students are able to learn facts and ideas: they are willing to memorize almost any reasonable amount of material. However, many of them lack the ability to use the facts and ideas so memorized. It is the author's contention that one of the major purposes of an examination is to determine proper rewards for the students who justly deserve them. If the examination tests primarily the student's ability to gather and to remember, rather than his ability to understand and interpret, the result is a meaningless assignment of rewards. Because the students are in college, their willingness and ability to memorize have already been demonstrated. Their very presence in class is, in a sense, a reward for having attained this willingness and ability.

It would appear that in an intellectual environment, examinations which test primarily the student's ability to use facts would lead to a more equitable assignment of merit. Nevertheless, when this is done the result is a grade which is generally much lower than that to which the student is accustomed. For example, the first hour examination of this kind, given each autumn, always yields a low median grade, usually between 50 and 55. It is significant that for the past seven years, the grades on this initial test have varied from 100 to 0, inclusive. Although subsequent examinations always produce several papers with scores below 20, the median grades are invariably higher. The median scores of the later examinations usually remain essentially constant.

There are certain types of questions and numerical problems which have been found useful in persuading the students to understand how specific laws and concepts can be applied. These are used almost exclusively; there are practically no questions on any

Based on a paper read at the 22nd Annual Summer Conference of the New England Association of Chemistry Teachers in Orono, Maine, August, 1960.

examination used during the year which can be answered by simple recall. For example, no definitions are ever requested.

On the first hour test one autumn, the students were asked

What is the atomic weight of Z if exactly 0.1 mole of X_8 and 40 g of Z_2 react completely to yield X_8Z_4 as the sole product?

Only 35 out of 345 students reported the correct answer although all had been warned that they were expected to know how to use atomic weights and interpret quantitative data. One disturbing aspect of this event was the fact that prior to taking the author's course over 200 members of the class had allegedly mastered what most of them called "weight-weight" problems; many of these students were disturbed to learn that their understanding of atomic weights and chemical equations had been incomplete. Others were more disturbed by what they called the "trick" question.

It is easy, for example, to determine which students understand the significance of atomic weights and chemical formulas by asking questions such as

How many grams of mercury(II) oxide, HgO, must be decomposed to yield half as many atoms as there are in 140 g of argon?

A November hour examination contained

Two-tenths of a gram of a gas occupies 100 ml at −136.5°C and 760 mm of Hg. What is the mass of one mole of the gas at 1520 mm of Hg and 0°C?

Nearly half the students who first obtained the mass of one mole then proceeded to "correct" the mass to that at 1520 mm. Robert Boyle must have turned over in his grave.

Two recent mid-year tests contained none of the conventional chemical symbols and names, either of elements or compounds. There were no atomic weights printed on the examination paper although each student did have a conventional periodic chart. Nevertheless, all the concepts that were "covered" on the semester hour tests were included in some way on these mid-year examinations.

One section of one of these included

Singly positive ions of element EM were present in the electrolyte of an electrolytic cell. The passage of 19,300 coulombs of electricity through the cell yielded 17.10 g of elemental EM. What is the atomic weight of EM? At which electrode did the EM appear? How many extranuclear electrons are in the uncharged atoms of EM?

A problem that appeared on a final examination was

An ionic compound A consists only of potassium ions and those of the conjugate base of a monoprotic molecule named psychic acid. If 50 g of A is added to water to yield 500 ml. of solution, the *p*H of the solution is 9.5 and the potassium ion concentration is 0.91 *M*. (At. wt. of K is 39.) If 18 g of psychic acid is added to pure water to yield 2.5 liters of solution, is the oxonium (hydronium) ion concentration equal to, less than, or greater than 0.1 *M*?

Approximately one-third of a class of 250 failed to realize that the fact implied definitely that psychic acid was a weak acid. For many more the molecular weight of the acid was particularly elusive.

One of the major stumbling blocks for many students seems to be a tendency to read carelessly, thereby failing to use correctly the facts presented in the questions. The author tries continually throughout the year to correct this tendency by pleading for strict interpretation of data and adherence to directions, and by including on each test at least four or five major questions and problems that require careful inspection of the printed word. Many of these are based on 100–200 word expositions either of situations or of specific physical and chemical phenomena. The questions and problems can be answered or solved, only by using the data given. Usually these data are facts unknown to the class prior to the test. The students are told to read carefully to find the key words in the statements, these words being either identical with or similar to those used in expressing some well-known law or concept. The application of the proper concept to the problem at hand then follows logically.

An October hour test presented

If the density of molybdenum is 10.2 g/ml, and that of aluminum is 2.7 g/ml, how many grams of molybdenum occupy the same volume as 81 g of aluminum?

Sixty per cent of the students in a class of 320 reported an erroneous value, although all had heard much about density in class and laboratory.

A November test contained a 195-word "story" about a situation regarding three reagent bottles. One of the statements within this exposition was

A tightly stoppered glass bottle *A* of 150-ml capacity contains 60 ml of liquid acetone. . . . What volume, in milliliters, inside bottle *A* is occupied by the substance acetone?

At least 150 students in a class of 350 concluded that the acetone occupied only 60 ml.

On a mid-year examination was

A sample of water is boiling briskly. What positive, correct statement can be made about the vapor pressure of the water?

In a group of 350 students, at least 175 blithely assumed, much to their subsequent chagrin, that the water was in an open vessel.

An April hour test presented

A student was given a clear blue aqueous solution *D*. Addition of dilute HCl to *D* yielded a white precipitate. Identify each ion that was definitely present in *D*.

Many students said that no definite statement could be made, obviously overlooking the fact that the solution was aqueous and that oxonium and hydroxyl ions were definitely present. A sizable group stated that copper(II) ions *might* be present, neglecting the fact they were asked which ions were *definitely* present. Others said that some ions of Group I were definitely present, failing to realize they were asked to *identify* each ion.

On a November test was

What mass of gaseous methane (mol. wt., 16) at 0.5 atm and 100°K contains the same number of molecules as 8.5 g of gaseous ammonia (mol. wt., 17) at 1 atm and 0°C?

Nearly half the students in a class of 350 failed to recognize that equimolar amounts (0.5 mole) of the two gases would contain the same number of molecules. Because the problem appeared to be an Avogadro's Law problem, many became hopelessly lost while computing the volumes of the gases. These students were shaken when they finally realized that only

two very simple divisions were needed.

While the organization of the course and its syllabus are important factors in determining the depth and breadth of a student's work, it is the examination program which best sets the pattern for his intellectual development. If he is rewarded most for his ability to memorize and reproduce from memory, this is the aspect of his study which will receive the greatest attention. If he is required continually to use facts and develop his own ideas on examinations, his attitude toward the course and toward studying for the course will be oriented in quite a different way. Good examinations teach the student as well as inform the instructor. Interestingly enough, questions which ask the students to *use* facts perform both of these functions better than any other kind.

GUEST AUTHOR
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Textbook Errors, 33

The Paddle-Wheel Crookes Tube

Many chemistry teachers commonly introduce the subject of atomic structure through a study of Crookes tubes.[1] One of these tubes has a small paddle-wheel which can be placed in the radiation, and the radiation then deflected one way or the other with an external magnet. The paddle-wheel rotates and its direction of rotation can be changed by deflecting the beam.

Professor Thomas Brown of the Department of Physics of Harvey Mudd College pointed out to the CHEM Study Writers Group in the summer of 1960 that this paddle-wheel experiment is not evidence for the mass of the electron, and that momentum transfer has little to do with the motion of the paddle-wheel. The effect is actually the same as that in a radiometer rotated by light. He referred us to the following quotation from "Conduction of Electricity Through Gases" (J. J. Thomson, Cambridge University Press, **1903**):

Mechanical effects produced by the rays. A secondary result of the thermal effects produced by the rays are the very interesting mechanical effects which have been especially studied by Crookes and Puluz. A typical example of these is afforded by the well-known experiment due to Crookes represented in Figure 163 (Crookes tube), where the axle of a very light mill with a series of vanes is mounted on glass rails, in a vacuum tube; when the discharge passes through the tube the cathode rays strike against the upper vanes and the wheel rotates and travels from the negative to the positive end of the tube.

A simple calculation will show that we cannot ascribe the rotation to the momentum communicated to the vanes by the impact of the corpuscles against them; for, take the case when the rays are so powerful that they carry the very large current of 10^{-8} amperes, and that they move with the very high velocity of 10^{10} cm/sec. If N is the number of corpuscles striking a surface in unit time, m the mass of the corpuscles; then supposing the corpuscles to rebound from the surface with a velocity equal to that with which they impinge against it, the momentum communicated to the surface in unit time is $2Nm10^{10}$. If e is the charge carried by a corpuscle, then Ne is the current carried by the rays, in our case 10^{-8} in absolute measure. Hence, the momentum communicated to the surface per second is equal to $2\ m/e\ 10^{4}$ dynes or, as $m/e = 10^{-7}$, to 2×10^{-3} dynes. This is equivalent to a difference of pressure, on the two sides of a vane 1 cm² in area of one-five-hundred-millionth part of an atmosphere; an effect altogether too small to explain the movement of a body such as that represented in Figure 163 (Crookes tube). This movement is probably due to an effect similar to that observed in a radiometer, as the impact of the cathode rays will make one side of the vanes much hotter than the other. Starke[2] has shown that when the vanes are arranged so that the radiometer effect is eliminated, the mechanical effect is exceedingly small—in his experiments, where the current carried by the cathode rays was 10^{-7} amperes and the potential difference 10,000 volts—certainly less than 10^{-4} dynes.

The effect in the Crookes paddle-wheel tube is therefore the same as in the light radiometer and should be interpreted in the same way. Excellent discussions of this latter effect may be found in most common textbooks on the kinetic theory of gases. A complete discussion is given in "Kinetic Theory of Gases," (E. H. Kennard, McGraw-Hill Book Company, Inc., **1938**). Briefly, the radiometer effect is due to the tendency of the gas to creep over a surface from a cold to a hot region. In a simple vane radiometer, the gas will creep around the edges toward the center of the heated or bombarded side. This effect tends to increase the gas concentration on the heated side somewhat and result in a slight excess pressure on this side, thus repelling the radiometer vane. This tends to produce a revolution away from the bombarded side, so that the bombarded side recedes from the bombarding particles.

Thus it seems clear that the paddle-wheel Crookes tube should not be interpreted in terms of straight momentum transfer but in terms of the radiometer effect as has been thoroughly explored early in the developments of this instrument, as indicated above. However, since almost no chemistry courses discuss the radiometer effect, it is apparent that the paddle-wheel Crookes tube should now be largely removed from any such course.

Suggestions of material suitable for this column and guest columns suitable for publication directly are eagerly solicited. They should be sent with as many details as possible, and particularly with references to modern textbooks, to Karol J. Mysels, Department of Chemistry, University of Southern California, Los Angeles 7, California.

[1] Since the purpose of this column is to prevent the spread and continuation of errors and not to evaluate individual texts, the source of errors discussed will not be cited. To be presented, the error must occur in at least two independent standard books.

[2] STARKE, H., *Ann.*, **308**, 101 (1900).

Chem Ed

BOOK REVIEWS

Experiments for Instrumental Methods

Charles N. Reilley, University of North Carolina, Chapel Hill and *Donald T. Sawyer*, University of California, Riverside. McGraw-Hill Book Co., Inc., New York, 1961. x + 412 pp. Figs. and tables. 22 × 28 cm. Paperbound. $5.95.

This laboratory manual, the first one of its kind in instrumental analysis, offers a refreshing new approach to experimental instrumentation. The manual develops a perspective which I hope will set the pace in a subject which more and more schools are introducing into their curriculum. (See for example THIS JOURNAL **37**, 279 (1960).)

The experiments are classified within five major categories: electrometric, optical, separation, radiochemical methods, and instrumentation. Experiments are devoted to the determination of equilibria data, e.g., ionization constants of phosphoric acid, acetic acid and bromthymol blue as well as the analysis of unknowns. Each topic has a short explanatory introduction. The experiments within a topic have additional reference sources to which the student usually must refer. The authors have sought to avoid the stultifying workbook approach by using a liberal dosage of questions interspersed throughout the text which require a synthesis of ideas and data to answer them.

Although there are a few familiar experiments which have been refined, there are a number of new experiments which are worth mentioning. The section on endpoint detection with various polarized electrodes is explained quite clearly and demonstrated with simple experimental examples. The effect of the controllable variables in a spectrophotometer are examined very intensively with the answers left to the student. "Precision" and "Differential" spectrophotometry are explored for determination of major components in an unknown. Tristimulus colorimetry is also treated. There is a rewarding section for the beginner in the use of infrared spectra for qualitative analysis. A very interesting use of gas chromatography in analyzing reaction mixtures of 1- and 2-butanol to study the mechanism of this elimination reaction is included under separation methods.

The last section, instrumentation, introduces in an understandable manner, electronics as it pertains to chemical instrumentation. There are experiments for construction and evaluation of some simple and useful equipment. The section ends with a theoretical trouble-shooting problem which I recommend to anyone who would like to teach students how to service their own electronic equipment.

My only reservation based on my own personal experience stems from the separation of the chemistry from the instrumentation. How much of a black box will a *p*H meter or spectrophotometer be to the student at the end of the course? The fact that the theory of instrumentation was treated is certainly a major step to avoid this. Experience will show if it is enough.

The cost of an instrumental analysis course is a primary consideration. The authors have kept this in mind in designing their experiments. The Spectronic 20 is used for most of the spectrophotometric experiments, and diagrams for constructing inexpensive electroanalytical equipment are included. Experiments using more expensive equipment are also included but they represent a minor portion.

The manual has 18 useful tables in the appendix. The figures and diagrams are clearly drawn. There appears to be sufficient attention paid to important details so that the experiments will work. It is an eminently *practical* laboratory manual. I recommend it with enthusiasm.

FREDERICK D. TABBUTT
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Textbook of Inorganic Chemistry

S. Young Tyree, Jr., University of North Carolina, Chapel Hill and *Kerro Knox*, Bell Telephone Laboratories, Summit, New Jersey. The Macmillan Co., New York, 1961. vii + 434 pp. Figs. and tables. 14.5 × 21.5 cm. $7.

The authors express the view that the recent trend in American universities in the first year course in chemistry is to place more emphasis on theory and less on the descriptive details of the variegated behavior of the elements. They have written a book, presuming only a background of general chemistry, which treats inorganic chemistry as a subject apart and in its own right. Only limited use is made of physical chemical principles, and where these are employed an introductory discussion is provided, e.g. use of the Nernst equation in relation to single electrode potentials. The treatment of the elements is more or less uniform, following a normal development based on the periodic table. Some of the more familiar elements are not discussed in appreciably greater detail than one might find in a standard general chemistry text. On the other hand, the chemistry of many elements scarcely mentioned in the standard first year treatment is discussed in as much detail as that of the more common ones.

With the increased emphasis on science and chemistry in many high schools, it appears to this reviewer that the well-trained student with high school chemistry could easily begin his college study of inorganic chemistry with a book such as this, perhaps bypassing most of the sketchy treatment of inorganic chemistry frequently provided in the usual general chemistry text.

The book is clearly written, appears to be accurate in factual detail, and suitable as a text for an undergraduate inorganic course. A useful appendix summarizing the rules of inorganic nomenclature is provided.

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Reviewed in This Issue

Charles N. Reilley and *Donald T. Sawyer*, Experiments for Instrumental Methods

S. Young Tyree, Jr., and *Kerro Knox*, Textbook of Inorganic Chemistry

Robert F. Chapman, Editor, Separation Processes in Practice

Keith M. Seymour, Organic Chemistry

Cyril Stanley Smith, A History of Metallography

F. A. Gunther and *L. R. Jeppson*, Modern Insecticides and World Food Production

Philip Pollack, Careers and Opportunities in Science

John Mitchell, Jr., I. M. Kolthoff, E. S. Proskauer, and *A. Weissberger*, Editors, Organic Analysis. Volume 4

R. Byron Bird, Warren E. Stewart, and *Edwin N. Lightfoot*, Transport Phenomena

Fritz Feigl, Spot Tests in Organic Analysis

Philip W. West and *Maurice M. Vick*, Qualitative Analysis and Analytical Chemical Separations

L. H. Ahrens and S. R. Taylor Spectrochemical Analysis

Jean Timmermans, The Physico-Chemical Constants of Binary Systems in Concentrated Solutions. Volume 4: Systems with Inorganic plus Organic or Inorganic Compounds (Excepting Metallic Derivatives)

Separation Processes in Practice

Edited by *Robert F. Chapman*, Atlantic Refining Company, Inc., Philadelphia, Pennsylvania. Reinhold Publishing Corp., New York, 1961. v + 209 pp. Figures. 13.5 × 19.5 cm. $4.95.

This volume is a collection of seven papers presented at a one day meeting in Philadelphia of the American Institute of Chemical Engineers. The prompt publication of these papers (and the ensuing discussion of each) following the March, 1960, meeting is in accord with the goal, stated in the preface, of supplying practical information for the young chemical engineer.

The first three chapters on Fundamentals of Mass Transfer Processes (Leon Lapidus), Fundamentals in the Design of Liquid Extraction Processes (W. G. Braun, R. H. McCormick, and M. R. Fenske), and Ion Exchange (R. L. Noison) have been approached in such a basic way that they will be as valuable to the young chemist as to the chemical engineer. Though each chapter is brief, the coverage is surprisingly broad.

The emphasis of the last four chapters clearly shifts to engineering considerations such as plant-scale equipment and typical breakdowns of production costs. Theoretical aspects are mentioned in a concise descriptive fashion.

Scale-up and Performance Relations for Continuous Crystallizers (W. C. Seaman) discusses the interrelationships between rate of seeding, rate of nucleation, and crystal size. Several equipment designs for removing fines are illustrated.

Preliminary Evaluation of Electric Membrane Processes for Chemical Processing Applications (W. E. Katz) treats the operating factors in a general way before examining in detail the concentration of sea water to produce salt brine. A similar approach has been used by E. G. Scheibel in his chapter, Separation of Aromatics and Paraffins by Solvent Extraction. In this case the author included a detailed discussion of solvent refining of gasoline with water.

The final chapter, Comparison of Tray Types in Fractionation Columns (R. S. Eagle), contains over a dozen photographs of different trays for promoting vapor-liquid contact in distillation columns. The brief comparison of the performances and costs with which the chapter concludes should be helpful to the young engineer to whom it was addressed.

The reviewer, who is a chemist rather than an engineer, believes that the meeting probably had a much more profound effect on the young engineers than will the written record of the proceedings. This conclusion in no way reflects on the authors, editor, or publisher, but reflects the realization that this volume will have to compete with established treatises on each of the subjects. Though the expensive treatises may lack some recent information their completeness may well prove to be an overriding factor.

L. B. Rogers
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Organic Chemistry

Keith M. Seymour, Butler University, Indianapolis, Indiana. Prentice-Hall, Inc., Englewood Cliffs, New Jersey, 1961. xiv + 321 pp. Figs. and tables. 16 × 23.5 cm. $6.75.

This text is written as an introduction to organic chemistry to provide an understanding of the field for students desiring no additional chemistry and to lay a foundation for those entering advanced courses. It is the belief of the author that "the needs of all students in their introduction to a subject are the same regardless how diverse and specialized their later needs may become."

The outline of this book parallels the pattern of many organic texts in which the introduction coordinates the concepts from the first year of chemistry, followed by a treatment of the hydrocarbons and their derivatives, and concluding with natural products. The aliphatic and aromatic series are separated although similarities and contrasts are indicated while presenting the aromatic compounds. To minimize the material to be mastered only the more frequently encountered preparations and reactions of the functional groups are given. There are some inadequacies, i.e., distinction between classes of amines with acid halides. There are over-emphases, i.e., proof of structure for the carbonyl group. To decrease the introduction of new ideas too rapidly it should be noted that reactions or preparations of a class of compounds is frequently deferred; i.e., no mention is made of the substitutionary reactions of the alkanes until the preparation of the alkyl halides. This may have significance by forcing a student to look for interrelationships between the several classes of compounds. Chapters 11 and 25 are excellent summaries of the cogent reactions. The interpretation of physical properties based on structural features is good. The problems require an application of knowledge rather than a direct quote from the text. The non-technical introduction of certain mechanisms and modern terminologies should be sufficient to make a student realize that the theorist is striving to interpret the observed facts. This seems to be the intent of the author who desires "to present no principle, no matter how important to a more advanced treatment of the subject, which is not usable by the student at his stage of development and his understanding of the science."

It is unfortunate that a new book has its mistakes, some of which do not detract from its usefulness, while other may leave an erroneous imprint on the mind of a student. There have been a number of discrepancies in nomenclature. Mixed ethers should be three words, as methyl ethyl ether and ethyl propyl ether (p. 71). The IUC ending for a glycol is -diol (p. 70). Several names are given in two words instead of one. If groupings within the names are to be emphasized, they can be hyphenated. Illustrative would be phenylhydrazine (p. 93, 270); phenylhydrazones (p. 93); benzenesulfonic acid (p. 185, 210); 1,2-ethanedicarboxylic acid (p. 223); benzenediazonium chloride (p. 224). The structure for the bisulfite addition to a carbonyl group should be C-S, not C-O-S (p. 93), as the correct name, hydroxy sulfonic acid (p. 94), would indicate. No hydrogen is relased in the formation of an osazone (p. 270). It is doubtful that thermal cracking of hydrocarbons produces higher homologues which could be inferred from the discussion (pp. 44–45).

The text is easy to read and the subject matter is presented in understandable terms. Topics, as rubber, plastics, sterols, and heterocyclics, normally found in a brief course have been omitted. Possibly, a student culminating his study of chemistry should have contact with these areas. On the other hand if a student is stimulated to seek out supplementary information, one can say "mission accomplished."

Bernard A. Nelson
Wheaton College
Wheaton, Illinois

A History of Metallography

Cyril Stanley Smith, Institute for the Study of Metals, University of Chicago. University of Chicago Press, Chicago, 1960. xxi + 291 pp. 110 figs. and 2 tables. 15 × 25.5 cm. $8.50.

Cyril S. Smith, formerly director of the Institute with which he is connected, has not only carried on important research in his field, but has had a long interest in the history of metallurgy and related fields which led to the translation of the classical works of Biringuccio and Ercker. His most recent historical venture leads to this fine study of an area that has been virtually untouched. Smith's meticulous scholarship, coupled with a superb publishing job, has produced a book that is both useful and beautiful.

The first section of the book deals with the metallography of such art works as Merovingian pattern-welded blades, Damascus blades, Japanese swords, etched armor, and welded gun barrels and swords. The author points out how armorers and jewelers took advantage of the structure of metallic crystals to produce artistic surface effects. Following a section on the corpuscular philosophy of the seventeenth century there is developed the efforts of early microscopists to gain an understanding of metals. Such studies were premature, however, neither the microscope nor corpuscular theory being capable of dealing with the problems involved. Most useful at the time was the study of fracture. Réaumur, in studying the changes associated with the conversion of iron into steel, became interested in the nature of fracture patterns and published a book dealing with the subject in 1724.

The real hero of the book is Henry Clifton Sorby (1826–1908) whose microscopic studies, starting in 1863, led to the scientific era in metallography. The painstaking cutting, polishing, and etching processes introduced by Sorby made it possible to relate the well-known properties of various forms of iron and steel to specific visible structural differences. The

(*Cantenued on page A634*)

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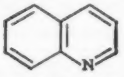

It was an exhilarating affair, particularly when we succeeded in pushing the yield close to 100%. Then we passed 100%, which was even more exhilarating. This we explained by assuming that the nitrobenzene was being reduced to more aniline for participation in the reaction. (Zdenko Hans Skraup would have been justifiably provoked with us for tortuous reasoning. His original proposal was merely to react nitrobenzene, glycerine, and sulfuric acid. Later, aniline was included.)

Anyway, we took pride in the efficiency with which we could furnish the world plenty of good, clean quinoline, a compound once considered the foulest of coal-tar derivatives.

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A free procedural abstract and the quinoline come from Distillation Products Industries, Rochester 3, N. Y. (Division of Eastman Kodak Company). Same address can supply reagents and procedural abstracts to test many things for many ingredients. There is no charge for a list of the abstracts and none for the abstracts themselves, but there is a slight charge for the reagents.

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BOOK REVIEWS

sequence of constituents which became evident as the carbon content of iron was increased led to a new level of understanding of the nature of steel.

The author brings out the retarding effect on the development of a theory of the metallic state which resulted from the rise of molecular concepts during the nineteenth century. While the concept of molecular aggregation was useful in understanding gases and even liquids, it delayed progress in understanding the solid state until the introduction of x-ray techniques established the lack of molecular organization in many solid substances. It is of interest to note that chemistry, particularly organic, profited enormously by use of molecular concepts in the nineteenth century. However, a theoretical construct which is useful in one area may do much damage when overextended.

The last part of the book deals with the contributions of Tschernoff in Russia, Martens in Germany, Osmond in France, Roberts-Austen in England, and H. M. Howe in America toward an understanding of the phases present in steel in relation to composition and heat treatment. Osmond, by combining microscopy and thermochemistry, converted metallography into a sound science. The book ends with a brief review of developments after 1890, particularly the application of thermodynamics by Le Chatelier, the application of Gibbs' Phase Rule by Roozeboom, the introduction of x-ray crystallography to metals by the Braggs, and the recent application of the electron microscope. The author has succeeded very well, not only in giving us a well-developed history of the subject, but in showing the interplay of disciplines and ideas in the history of science.

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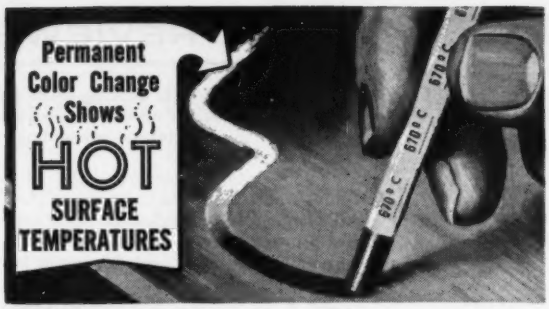
Modern Insecticides and World Food Production

F. A. Gunther and *L. R. Jeppson*, Citrus Experiment Station, University of California, Riverside, California. John Wiley & Sons, Inc., New York, 1960. xv + 284 pp. Figs. and tables. 15.5 × 23 cm. $8.50.

The introductory chapter of this book deals with insects and mites, and their plant and animal hosts. The coverage of this vast subject in one chapter of only 19 pages is necessarily sketchy though it must be admitted that the authors have chosen their material with good judgment. It is felt, however, that the reader should have considerable entomological knowledge before he is fully able to digest the chapter content.

The following 15 chapters deal with the methods, the tools (machinery and chemicals) and the problems resulting from insecticide usage. Chapter 6 which presents the tremendously important problem of deposits and residues is well done in that it assembles and summarizes much

(*Continued on page A636*)



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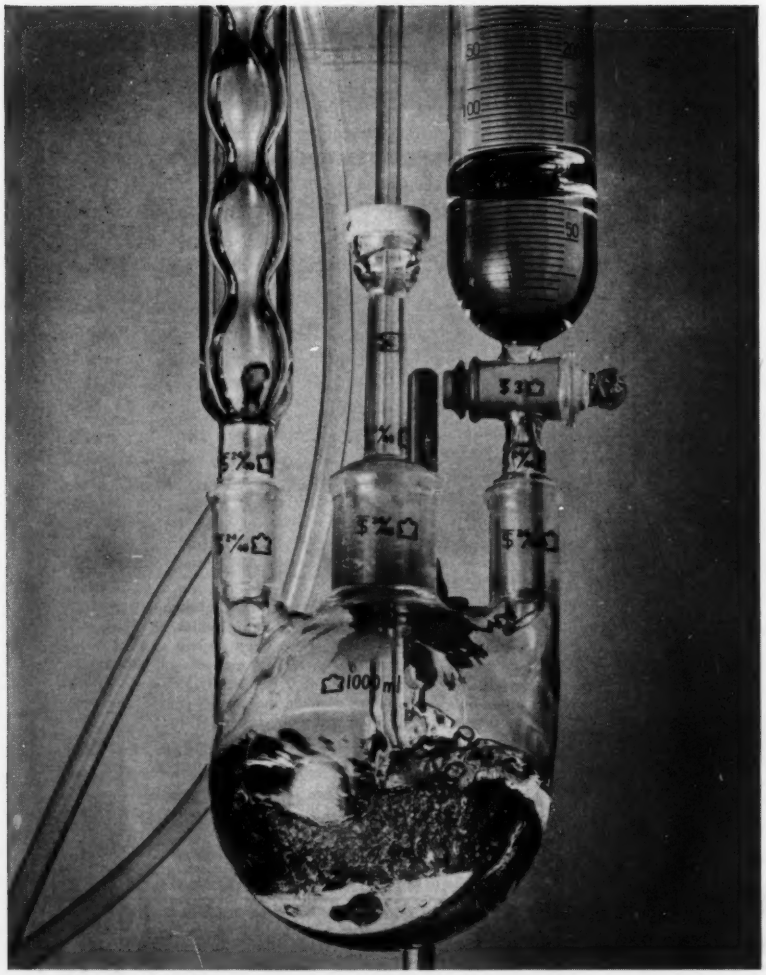


BOOK REVIEWS

widely scattered data dealing with this subject. The tables of this chapter also contribute to a better understanding of the problem.

Also of vital importance is the subject of resistance of insects and mites to chemicals which is presented in summarized form in chapter 7. Again this is a clear presentation, but it probably could have been enlarged to advantage. Certainly some of the points in this chapter deserve a more extended treatment.

Another excellent chapter is headed Modern Insecticides and in it there are presented data on the importance of insect and mite control which should be of special interest to producers and consumers alike. Chapters which deal with the insecticide groups are fairly complete, carrying a vast amount of information in summarized form.

In the preface the authors state that the book was not written for specialists in the field of economic entomology or the chemistry of insecticides. This, in the opinion of the reviewer, would place decided limits on the usefulness of this volume, as it is felt that very few laymen could read the book with perfect understanding. At least it will not hurt the specialist to read it.

The volume is well illustrated, well bound and printed, and textual mistakes are very few. Overall it is an excellent review of a big field.

C. R. Cutright
Ohio Agricultural Experiment Station
Wooster

Careers and Opportunities in Science

Philip Pollack. E. P. Dutton & Co., Inc., New York, 1960. 194 pp. Illustrated. 14 × 21 cm. $3.95.

A considerable amount of material is available today for the expressed purpose of guiding young people in the selection of a career. This revised edition of a similar effort published in 1954 "is concerned with the work of scientists of all kinds, but especially of those engaged in basic research."

The author wisely elects to consider only a limited number of scientific fields. These include physics, chemistry, biochemistry, atomic energy, geology, biology, medical research, and conservation. However, within the individual areas, many different types of jobs are discussed which, in effect, broadens the coverage considerably. Discussion includes opportunities in industry, education and government. In the presentation of these areas much use is made of current discoveries; how they were achieved, and the present frontiers. These discussions alone should prove to be very informative and intriguing to the student. The reader is then introduced to actual job situations which will most likely be encountered in the field. No attempt is made to overglamorize a particular career. The disadvantages, hazards, and opportunites of the various

(*Continued on page A638*)

DETACH PAGE BY CUTTING ALONG THIS LINE—INDIVIDUAL DEMONSTRATIONS MAY BE CUT APART AND PASTED ON 5 × 8 CARDS

Displacement Reactions—Spongy Tin

A Chem Ed TESTED DEMONSTRATION

Submitted by: Fidel Villarreal and Octavio Garcia, Institute Technológico de Monterrey, Monterrey N. I., Mexico
Checked by: H. Ann Schwartz, Bucknell University, Lewisburg, Pennsylvania

PREPARATION

Provide two 600-ml beakers, stirring rod, and granular zinc (40 mesh). Prepare a 10% solution of tin(II) chloride and 6 *M* hydrochloric acid.

DEMONSTRATION

Pour 200 ml of $SnCl_2$ into the beaker and add 40 ml of 6 *M* HCl. Stir the solution. Cover the bottom of the beaker with 2 to 3 g of granular zinc. After a few seconds spongy tin rises to the surface of the liquid.

REMARKS

Hydrogen and tin are formed by the following reactions:

$$Zn + 2HCl \rightarrow H_2 + ZnCl_2$$

$$Zn + SnCl_2 \rightarrow Sn + ZnCl_2$$

Hydrogen bubbles prevent the tin from settling to the bottom of the beaker. The sponge can be removed from the beaker in one piece with a glass stirring rod. The experiment may be repeated ten times or more using the original solution. Concentrated hydrochloric acid and a few crystals of tin(II) chloride may be added to the original solution if more sponges are desired.

DETACH PAGE BY CUTTING ALONG THIS LINE—INDIVIDUAL DEMONSTRATIONS MAY BE CUT APART AND PASTED ON 5 X 8 CARDS

Electrode Potentials with a Vacuum Tube Voltmeter

A Chem Ed TESTED DEMONSTRATION

Submitted by: Louis Gachic, Regis College, Denver, Colorado
Checked by: J. R. Brandou, Michigan State University, East Lansing

PREPARATION

Prepare 1 × $^1/_2$-in. sheet or heavy foil samples of the metals Zn, Cu, Pb, Ag, and Sn and clean with steel wool. Prepare 1 *M* solution of soluble salts of each metal and $NaNO_3$ (e.g., $CuSO_4$, $Pb(NO_3)_2$, $SnCl_2$, $ZnSO_4$, $AgNO_3$). Cut an 11-cm filter paper as shown in the figure and lay it on a 6-in. square glass plate. Connect a vacuum tube voltmeter to 110-v ac and set to 1.5-v dc range.

DEMONSTRATION

Place 2 drops of 1 *M* $CuSO_4$ solution in one of the areas on the filter paper and lay the piece of copper on the moistened paper. Repeat the procedure for $ZnSO_4$ and Zn, $SnCl_2$ and Sn, and $PbNO_3$ and Pb (or $AgNO_3$ and Ag) in the remaining sections. Add a drop or two of $NaNO_3$ to the central area to act as a salt bridge between the metal-metal ion sections. Check the potentials between pairs of the metals with the vacuum tube voltmeter and record the readings.

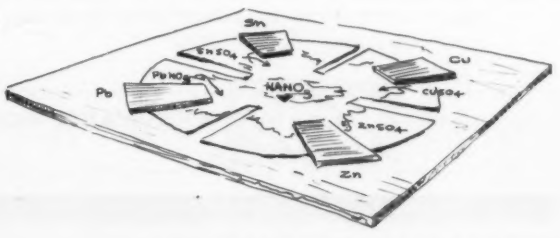
Concentration cells may be demonstrated by comparing the voltage produced when one of the metals is in contact with a solution of its salt at a concentration considerably different from 1 *M*.

REMARKS

At the start of the experiment two samples of the same metal should be used in contact with the same solution of the metal salt, to show that no potential difference is indicated by the voltmeter. The $NaNO_3$ solution can be effectively used as an extremely dilute metal ion solution for concentration cell demonstrations.

Comparisons between calculated and observed voltages are given in the table:

	Zn-Sn	Zn-Cu	Sn-Cu	Pb-Cu
Observed	0.56	1.1	0.56	0.53
Calculated	0.62	1.1	0.48	0.46



careers are discussed in a clear and precise manner. The advantages and rewards, both tangible and intangible, are likewise clearly presented. Educational requirements are stated in terms of definite courses and years of training in order to qualify for the opportunities which may be available in the field at the various levels.

A very important chapter is devoted to the opportunities for women in the sciences. The contributions of women to science, both past and present, serve as illustrations of the definite and increasing place of women in this field. The author does not overlook the unique problems which will be encountered, but hastens to discuss the changing view of society toward women scientists.

An extremely helpful appendix contains tables of average salaries which presently prevail in the field. The tables are specific for the various fields. Sources of additional information concerning each field are listed. Another unique service is a listing of colleges and universities which train persons in the various specific fields.

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American Chemical Society
Washington, D.C.

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Organic Analysis. Volume 4

Edited by *John Mitchell, Jr., I. M. Kolthoff*, University of Minnesota, Minneapolis, *E. S. Proskauer*, Interscience Publishers, Inc., New York, and *A. Weissberger*, Eastman Kodak Co., Rochester, New York. Interscience Publishers, Inc., New York, 1960. vii +429 pp. Figs. and tables. 16 × 23 cm. $13.50.

This book is number four in a series of collective volumes which eventually will cover the entire field of organic analysis. The first of the series appeared in 1953; it, and two subsequent volumes, dealt largely with functional group analysis.

The bulk of volume 4 is devoted to instrumental analytical techniques. It contains six chapters: 1) Determination of Organic Peroxides, 2) Enzyme Analytical Reactions, 3) Gas Chromatography, 4) Application of Nuclear Magnetic Resonance Spectroscopy to Organic Analysis, 5) Crystallographic Methods of Analysis: X-Ray Diffraction and Microscopy, and 6) Application of Differential Thermal Analysis to High Polymers.

The treatment of organic peroxides is comprehensive, covering volumetric, colorimetric, and instrumental methods of peroxide estimation and concluding with recommendations for specific types of peroxides and comparisons of available methods. An extensive bibliography is provided, and, as in previous volumes, critical evaluation and comparisons of methods are made throughout the text. In some cases, improved procedures, differing somewhat from those in the original literature, are described. The discussion is written with authority.

The chapter on enzyme analytical reactions is a very brief survey of enzyme reactivities with a few specific analytical applications.

The important and rapidly developing field of gas chromatography is treated extensively in the longest chapter in the book. Although "the purpose of this chapter is to acquaint chemists with the potential of gas chromatography for organic analysis," the authors accomplish more than this. A considerable amount of general information is provided, in addition to the analytical applications. The concluding pages contain a series of tables in which are tabulated diverse specific applications, ranging from the accumulation of thermodynamic and kinetic data through application to foodstuff chemistry. Descriptions of work and instrumentation done in the authors' laboratory augment the abundant recent literature in the field.

There are available a number of broad treatments of the relatively new technique, NMR spectroscopy. In the present chapter the stress is on applications of high resolution spectroscopy to structure determination of organic molecules; neither the theory nor instrumentation are discussed as fully as in some of the other treatments.

Crystal optics and morphology, X-ray diffraction analysis, and fusion methods of analysis are the subject of Chapter 5. Discussions of principles and techniques

(*Continued on page A640*)

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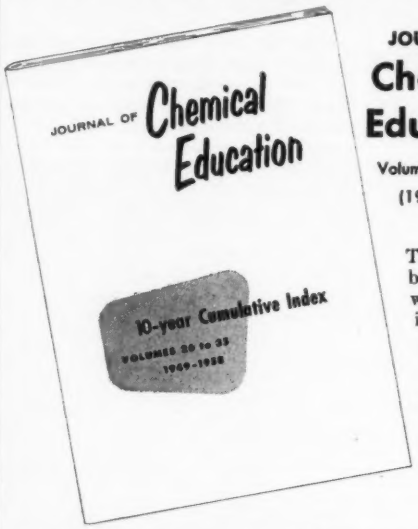

JOURNAL OF Chemical Education

Volumes 26 through 35 (1949–1958)

This index has been prepared with the special interests of the J.-Chem.-Ed. user in mind. Not only are the conventional entries made by author, title, and inverted title, but many suggestive subject listings have been used. For example, reference to an article entitled "Infrared Spectra of Chemisorbed Molecules" can be found under "acetylene," one of the compounds discussed. Preparation of the index involved hundreds of hours of careful checking not only of annual indexes, but the original articles for subject content.

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BOOK REVIEWS

are limited here by the space allotted to this large topic, but the chapter serves as a survey and introduction to analytical crystallographic methods.

The old technique of differential thermal analysis has only recently been recognized as a useful analytical tool for characterizing high polymers, although it has been used for some years in studying their thermal behavior. The author of the last short chapter in the present volume surveys the basic principles involved in the measurements, the equipment needed, and the procedures employed.

Some of the topics treated in the present volume are of such scope that it is difficult, or impossible, to do justice to them within the confines of a collective volume. However, the techniques presented here are increasingly important to the analytical chemist and demand treatment in any work aimed at a coverage of the field of organic analysis. At the least the discussions, with their ample bibliographies, can serve as a 'jumping-off' point for the novice in any of the fields.

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Transport Phenomena

R. Byron Bird, Warren E. Stewart, and *Edwin N. Lightfoot,* all of the University of Wisconsin, Madison. John Wiley & Sons, Inc., New York, 1960. xxi + 780 pp. Figs. and tables. 16.5 × 23.5 cm. $13.75.

Although there has been considerable change in engineering technique and practice since the pioneering work of Walker, Lewis, McAdams, and Gilliland was first published, textbooks dealing with the unit operations have adhered to the format established by these authors. The book, "Transport Phenomena," offers a refreshing and gratifying deviation from this format. This text is not a unit operations book in the classical sense in that the authors have not endeavored to encompass the scope of subject matter normally presented in such works. Instead the material covered in this text is limited to those processes which are basic to all unit operations—the transport of momentum, heat, and mass. Although this subject matter has been considered by many authors, "Transport Phenomena" derives its great value from the clever organization and convincing presentation of the material. This work is evidently a product of considerable classroom experimentation and the reader is stimulated by the clear exposition of the fundamentals of the processes studied, by the example problems which are carefully selected to illustrate the principles, and by the challenging problems which augment each chapter.

The book is arranged so that momentum transfer, heat transfer, and mass transfer are each considered in separate sections. However, each of these subjects is treated in an identical manner to stress the similarity in the mechanisms of these transport processes. The first chapter of each section defines the basic transport property (viscosity, thermal conductivity, or diffusivity) and describes methods of estimating these parameters when data are not available. In the following chapter the concept of a "shell balance" is introduced. This balance is simply a statement of the appropriate conservation law and is applied to systems which are easily visualized so that the reader can readily match the theoretical result with his experience. The shell balance also demonstrates the construction of a mathematical description of physical problems. This latter facet is an exceedingly important by-product since mathematics is the language of science and engineering.

After developing these fundamental concepts, the equations of change are derived. These equations represent transport in an arbitrary continuum. Examples are provided to illustrate the application of these relationships. In the following two chapters of each section, more complicated problems are considered (involving two or more independent variables) and the concept of turbulence is introduced. These topics are advanced for the beginner and would not be treated in the same detail in an introductory course.

Finally, the well established empirical relationships normally used by practicing

(Continued on page A641)

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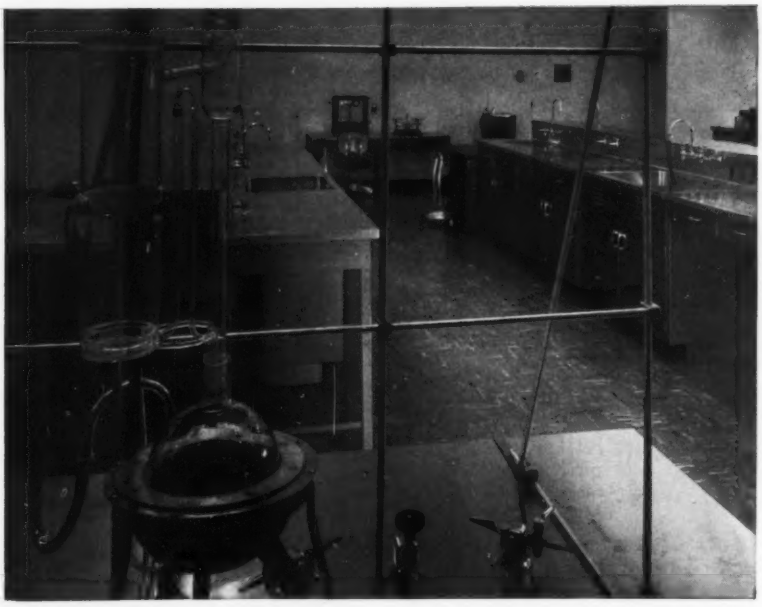

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BOOK REVIEWS

engineers in solving transport problems are coupled with overall (macroscopic) balances to resolve practical problems. This treatment is effective because the reader has been alerted to the necessity of these approximations and to the basis of the empirical relationships (usually in the form of a dimensional analysis).

As previously noted, "Transport Phenomena" is unique in its mode of presentation, in its depth of coverage, and in its fundamental approach. Undoubtedly, this text will be widely adopted for classroom use and will serve as a standard in the development of chemical engineering texts in other areas.

ROBERT S. SCHECHTER
University of Texas
Austin

Spot Tests in Organic Analysis

Fritz Feigl, University of Brazil. Translated by *Ralph E. Oesper*, retired, University of Cincinnati, Ohio. Elsevier Publishing Company, 1960. Distributed in the U. S. by D. Van Nostrand Co., Inc., Princeton, New Jersey. xx +675 pp. Figs. and tables. 16.5 × 23 cm. $13.25.

Spot tests for organic compounds were first presented only briefly in the earlier editions of Feigl's "Spot Tests"; and it was not until the fourth edition appeared in 1954 that this subject assumed sufficient importance to be treated in a separate volume, which was given the title, "Spot Tests in Organic Analysis." So great was the interest in this comparatively new field of analysis that two years later, in 1956, a new and enlarged fifth edition containing much new material was published; and now in 1960 a sixth edition has appeared.

As a result of extended studies prompted by the wide acceptance of the 1956 edition, Professor Feigl has introduced many new procedures based on pyrolysis reactions. He has shown that many such reactions lead to the formation of products which can readily be detected with high sensitivity, and which are at the same time characteristic for certain starting materials. Such reactions include hydrolytic and ammonolytic cleavages, displacement, condensations, and redox reactions which occur when organic compounds are melted or sintered with suitable reactants. A careful study of these new or comparatively unknown reactions has resulted in the discovery and development of many sensitive and strikingly simple tests. This has been the essential justification for preparing a new edition.

The arrangement of the material is essentially the same as in the previous editions. The description of numerous procedures and the chemical principles upon which they are based, together with extensive literature references, are presented. The first chapter deals with the development, present state, and prospects

(*Continued on page A642*)

of organic spot test analysis, and by reference to the more recent findings indicates the guiding principles which have proved useful in the search for new tests. There has been no significant change in Chapter 2 on Spot Test Techniques. Chapter 3 on Preliminary (Exploratory) Tests has been expanded considerably, and the number of sections increased from 25 to 32. The number of tests for functional groups (Chapter 4) has been increased from 49 to 70, and the number of tests for individual compounds (Chapter 5) has been increased from 78 to 133. The applications of these methods to the testing of materials (Chapter 6) has been approximately doubled with 111 sections, instead of 62. Altogether, this new edition describes and discusses nearly 600 tests in contrast to 386 in the 1956 edition.

In order to prevent the new edition from becoming too large the author has resorted to rather extensive deletions, abridgements, and typographical simplifications. In spite of this the size of the book has increased from 616 to 675 pages.

Perhaps no chemist has a greater knowledge of chemical reactions than Professor Feigl, and this knowledge is clearly reflected in the material presented in this book. It should be emphasized that it is more than a manual for the practical identification of organic compounds, although it brilliantly fulfills this function. The reactions, and the principles upon which these reactions are based in applying them as spot tests, are fully presented with great clarity.

Accordingly, this book should be of wide interest because it frequently provides valuable suggestions for non-analytical studies. It is designed not only for professional analysts, organic chemists, biologists, and forensic chemists who are concerned with solving analytical problems on a micro-scale, but also for teachers of chemistry. Organic chemistry laboratory courses have especially been considered.

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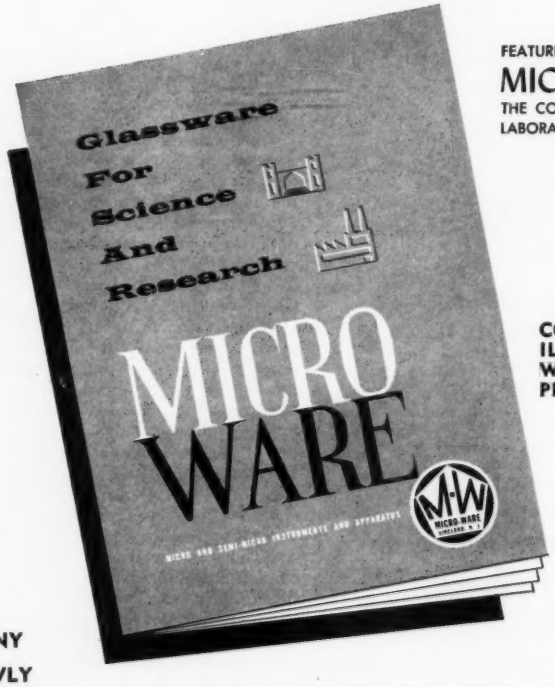

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Qualitative Analysis and Analytical Chemical Separations

Philip W. West and *Maurice M. Vick*, both of Louisiana State University. 2nd ed. The Macmillan Company, New York, 1959. xv + 302 pp. 22 figs. 14.5 ×21.5 cm. $4.50.

The second edition of this text is aimed at both the sophomore course in qualitative analysis and the second semester studies of the same subject incorporated in a general chemistry course for freshmen. For greater usefulness at the freshman level, some subjects were treated in more detail than in the first edition: Part I on experimental procedures was revised to take into account several years' practical experience with the new separation scheme and enriched with a new section on spot tests. Part II on fundamental theory is essentially unchanged. A whole 60-page section was added and forms the new part III of the book on metals, metallurgy, and reactions of the metal ions. Also added were sections on mathematical operations.

The outstanding merit of this text is unquestionably the detailed presentation of a new and workable cation separation scheme which is radically different from the traditional 110-year old Bunsen-Fresenius method of sulfide separation. After a chloride precipitation of two cations follows a "basic benzoate group" i.e. a controlled-*p*H precipitation of six cations with oxidation state 3 or 4, which is a much improved version of the time-honored basic acetate separation. Then, with the *p*H still on the acid side, a "fluoride group" of five cations is separated followed by an alkaline precipitation of seven cations as a "non-amphoteric group," leaving in a final solution three amphoteric ions.

A most valuable addition to the systematic separation scheme are the sections on reactions of individual cations which one finds throughout part I of the book, one for each cation. They are written in form of experiments to be made with knowns, interspersed with some exquisite little spotlights on inorganic chemistry and on special methods of analytical chemistry, which testify to the authors' rich experience in this field.

(*Continued on page A644*)

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The list of solutions in the appendix would gain in usefulness if gram solute per liter solution were given besides molarity. The format of the book is very handy, print is clear and easy to read, and illustrations are well done.

RICHARD E. FRANK
University of North Dakota
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Spectrochemical Analysis

L. H. Ahrens and *S. R. Taylor*, both of the University of Cape Town, South Africa. 2nd ed. Addison-Wesley Publishing Company, Inc., Reading, Massachusetts, 1961. xxiii + 454 pp. 16 × 23.5 cm. $15.

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Jean Timmermans, Universite Libre, Brussels, Belgium. Interscience Publishers Inc., New York, 1961. 1332 pp. Tables. 19 × 24.5 cm. $39.

This is the final volume of the series. In addition to "Systems with Inorganic plus Organic or Inorganic Compounds," (629 pages on water solutions), there are formula indexes, both organic and inorganic, and the bibliography. These latter cover the entries for all four volumes. A brief list of errata is included in the bound text. See THIS JOURNAL, **37**, A46, A384 (1960).

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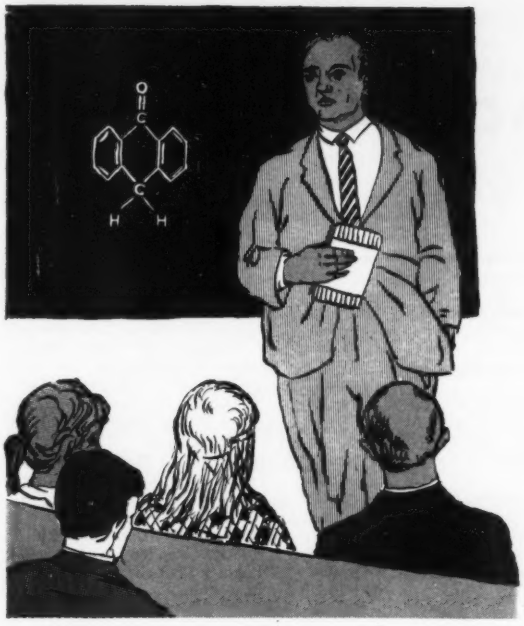

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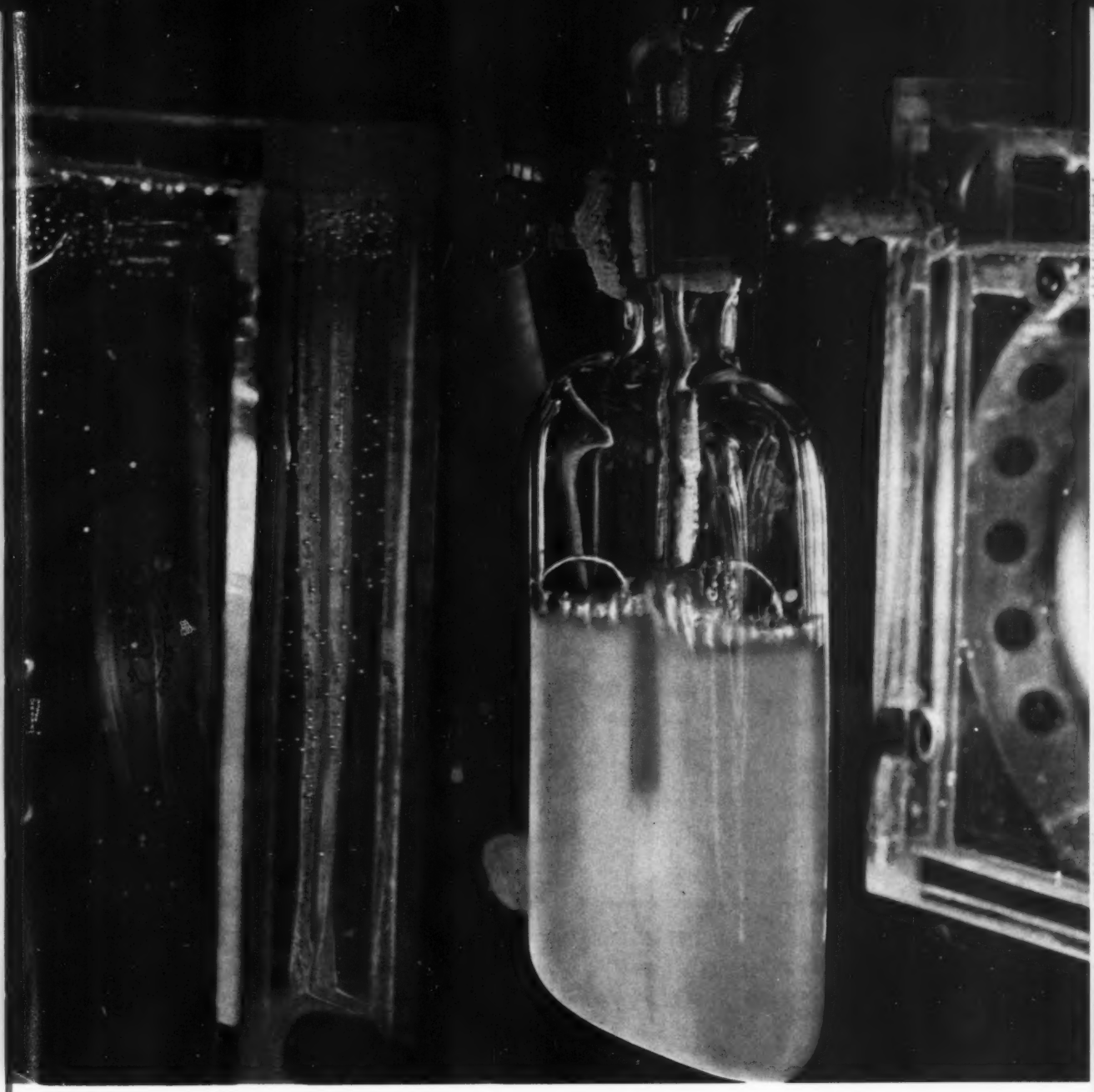

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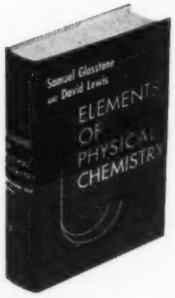

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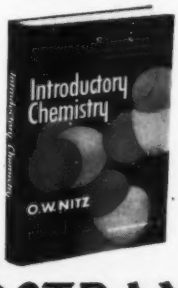

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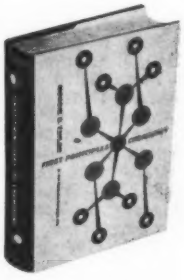

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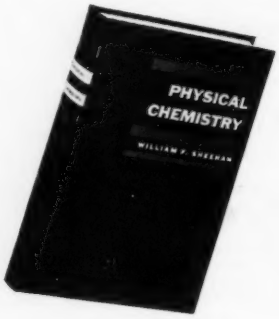

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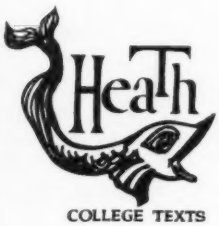

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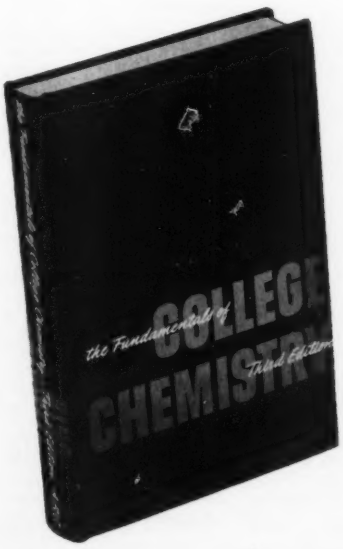

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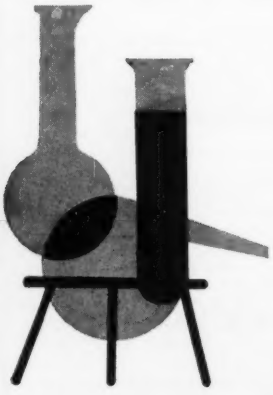

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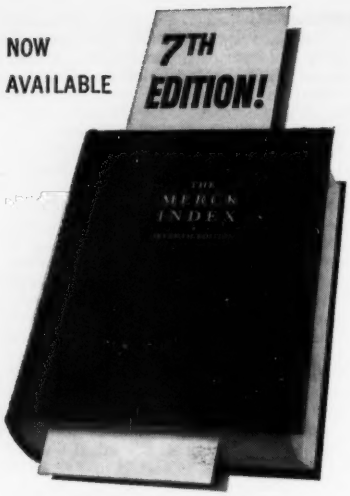

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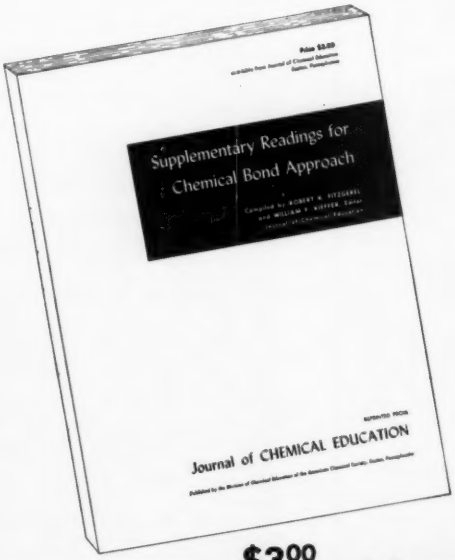

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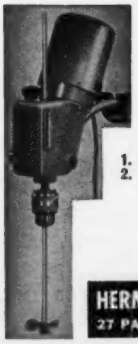

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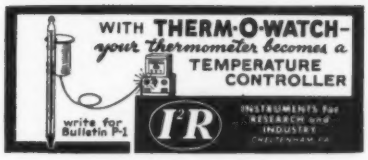

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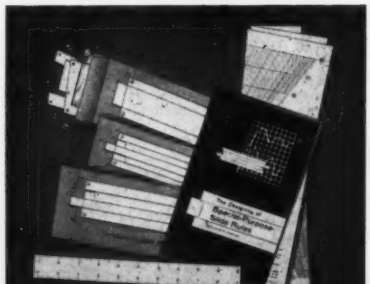

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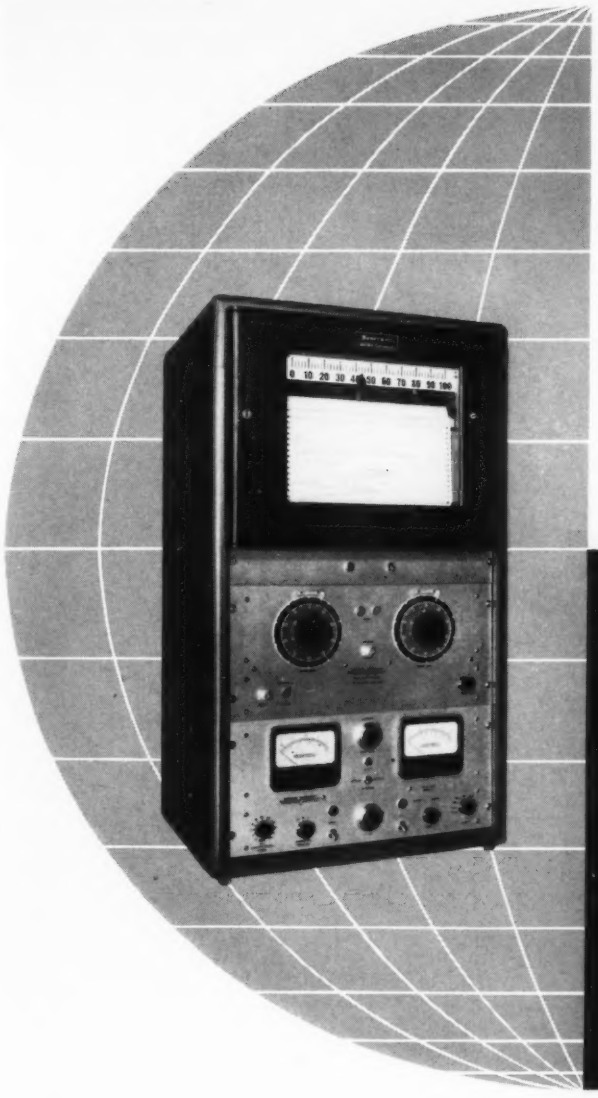

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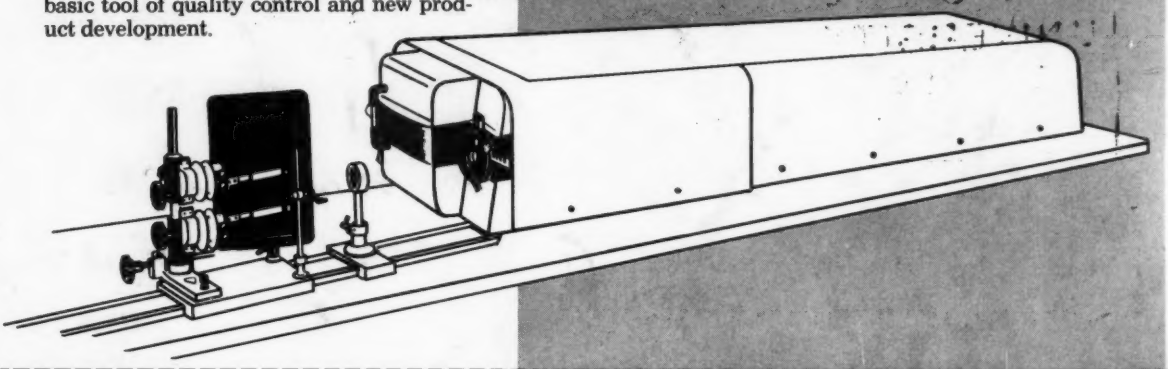

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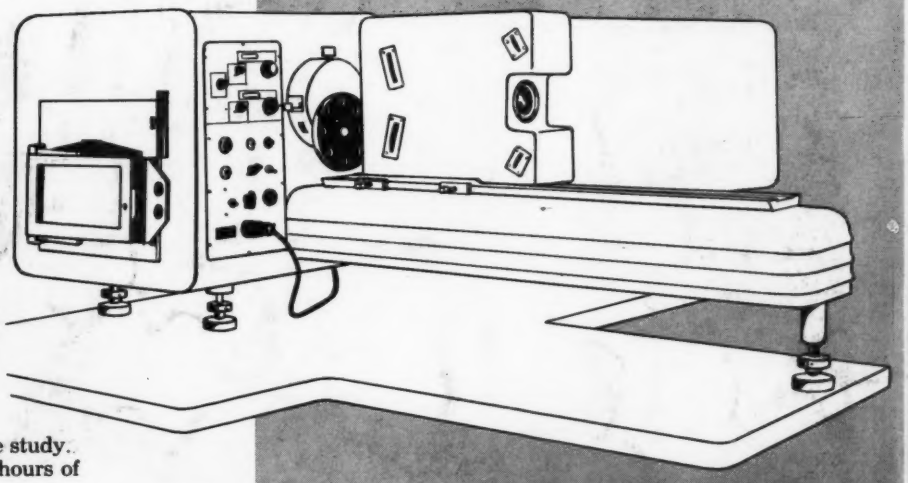

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